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EVALUATION OF THE INTEGRATION OF THE SANDWELL CENTRAX TM PROCESS INTO AN INTEGRATED TWO-STAGE LIQUEFACTION PROCESS

PHASE III

FINAL REPORT

by

Sandwell Technologies

MICROMEDIA

Work on this project was conducted under the auspices of the Canada Centre for Mineral and Energy Technology, Energy, Mines and Resources Canada.

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- Electric Power Research Institute - provided pilot plant size samples for testing,
- Orenda - superior capabilities in balancing and assembly,
- Horn Industries Ltd. - precision machining of the parts for the Centrax Separator,
- DSMA International Inc. - engineering services related to the details of the mechanical design aspects of the Centrax.

In addition to the contributions of these companies, the efforts of the Sandwell Technologies Staff are recognized in that they carried out the overall design/ overall management/ on site assembly/ testing of the first prototype Centrax separator pilot plant.

TABLE OF CONTENTS

CONFIDENTIAL

ACKNOWLEDGEMENTS

SUMMARY/SOMMAIRE

1.0 INTRODUCTION 1.

2.0 CENTRAX PILOT PLANT 3.

  2.1 Installation and Assembly 3.

  2.2 Process Description 3.

    2.2.1 Operation of the Centrax Separator 3.

    2.2.2 Operation of the Centrax Pilot Plant 3.

    2.2.3 Operation and Control of Solids Scraper 12.

3.0 COMMISSIONING TROUBLE SHOOTING/RUN HISTORY 19.

  3.1 Commissioning 27.

  3.2 Trouble Shooting/Run History 28.

4.0 SEPARATION PERFORMANCE; PRELIMINARY RESULTS 34.

  4.1 Objective of Test Program 34.

  4.2 Experimental Program 35.

  4.3 Typical Run Procedure 35.

  4.4 Test Results 36.

    4.4.1 Ash Content of Separated Liquid 36.

    4.4.2 44.

    4.4.3 46.

  4.5 Processing Performance 50.

5.0 UPGRADING OF SEPARATED PRODUCT 51.

  5.1 Description of Equipment 51.

  5.2 Testing Procedures 54.

    5.2.1 Feedstock Preparation 54.

5.2.2 Catalyst Preparation 54.

5.2.3 Testing 54.

5.2.4 Product Recovery 55.

5.3 Analytical Procedure 57.

5.3.1 Material Balance Calculation 57.

5.3.2 Analysis of Feedstocks 57.

5.3.3 Analysis of Products 57.

5.4 Results and Discussion 58.

6.0 ECONOMIC IMPLICATIONS OF CENTRAX DEASHING 62.

6.1 Economic Modelling - Coal Liquefaction 62.

6.1.1 Economic Modelling Methodology 62.

6.1.2 Model Description 63.

6.1.3 Cases for Comparison 64.

6.1.4 Model Documentation 65.

6.2 Model Assumptions 66.

6.2.1 Technical Assumptions 66.

6.2.2 Operational Assumptions 67.

6.2.3 Pricing Assumptions 67.

6.2.4 Capital Cost Assumptions 68.

6.2.5 Financing Assumptions 69.

6.3 Model Predictions 70.

6.3.1 Economic Significance of Centrax Deashing 70.

6.3.2 Sensitivity to Capital Cost 71.

7.0 DEVELOPMENT PLAN - PROTOTYPE TO EXTENDED TIME TRIALS 73.

7.1 Scope of Work 75.

7.2 Project Schedule 80.

7.3 Cost Estimate 80.

7.4 Program Implementation 86.

LIST OF TABLES AND FIGURES (Cont'd)

TABLES PAGE

4.1 Feed and Extractant Composition 34.

4.2 Run 7 Separated Liquid Concentration - Pilot Plant versus Laboratory Separations 43.

4.3 Run 7 Separated Solids Composition - Pilot Plant versus Laboratory Separations 48.

5.1 Experimental Condition 58.

5.2 Material Balance Calculation 58.

5.3 Properties of V110 Bottoms Run 247 59.

5.4 Properties of Liquid Feeds 60.

5.5 Properties of Products 60.

6.1 Capital Cost Factors 69.

7.1 Centrax Process Equipment Development Status and Goals 75.

7.2 Project Schedule 81.

7.3 Cost Summary Table 82.

7.4 Estimated Labour Cost ($CDN) 83.

7.5 Estimated Disbursements ($CDN) 84.

7.6 Estimated Cost by Task 85.

CONFIDENTIAL

LIST OF TABLES AND FIGURES

FIGURES

FIGURES	PAGE
2.1 Centrax Piping and Instrument Diagram	4.
2.2 Frontal View of Centrax Pilot Plant	5.
2.3 Side View of Centrax Pilot Plant	6.
2.4 Schematic of Centrax Separator	7.
2.5 Insulated Top Section of Centrax Separator	8.
2.6 Side and Back View of Centrax Pilot Plant	14.
2.7 Gravity Feed Into Centrax	15.
2.8 Centrax Deashing Process - 3 Stage Counter - Current Arrangement	17.
2.9 Hydraulic Power Pack	21.
2.10 Hydraulic Schematic	22.
2.11 Externally Mounted Hydraulic Controls & Indicators	23.
2.12 Pusher Control Schematic	25.
4.1 Ash Content Separated Liquid G Force - Feed Rate	37.
4.2 Ash Content Separated Liquid Type of Feed - Feed Rate	39.
4.3 Run 10 Ash Content - Scraper Operation	41.
4.4 Typical Cross Section of Formed Cake	47.
4.5 View of Cake of Establish Cake on Bowl Wall	48.
4.6 Close Up View of Liquid Overflow Ports	49.

LIST OF TABLES AND FIGURES (Cont'd)

FIGURES

5.1 Schematic Diagram of Hot Charge/Discharge 53.

5.2 Product Streams 56.

6.1 CSD Deashing Case A 64.

6.2 Centrax Deashing Case Hydrogen by Gasification Case B-1 65.

6.3 Centrax Deashing Case Hydrogen by Gas Reforming Case B-2 65.

6.4 Sensitivity Analysis - Economic Return vs. Centrax Capital Cost 72.

7.1 Example of Centrax Flow Diagram Process Scheme 1 (Wilsonville Run 243 Feed) 74.

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Summary

Covered in this report are the commissioning and preliminary testing of the pilot plant Centrax separator, prototypes 1. This work is denoted as Phase III of a three phase program. The laboratory process development for this patented separation process is described in Phase I, while the details of the hardware development of a separation apparatus for affecting the unique separation requirements are described in Phase II.

The Centrax separator was specially designed for the separation of solids from viscous hydrocarbon slurries. The essential features of the prototype unit are that:

- the processing part is, in essence, insolated from the drive section,
- separation of solids is affected independently of their physical properties.
- the requirement of inter-transport of solids in a multi-stage configuration has been eliminated.

The testing program revealed that the operating performance of this first prototype was outstanding in that it performed according to specifications. Operation at the relatively high speed of 4600 RPM and at the high temperature of 180°C presented no problems. Runs were terminated when the feed was exhausted, not because of operational difficulties. Throughout, the test program improvements were made in instrumentation, temperature control, and in the operation of the bottom bearing of the Centrax separator. Design changes to this bearing assembly are temporary. It needs to be redesigned for subsequent units.

Sommaire

Ce raport comprends la mise en marche et les essais préliminaires du prototype numero 1 de l'unité pilote de separation Centrax. Ce travail fut designé comme IIIemme Phase du programme. Le développement du procedé au laboratoire fut decrit dans la Iere Phase tandis que les détailles du développement des aspects mechaniques de l'appareil de séparation capable de satisfair les demmandes uniques de separation furent decrites dans la IInde Phase.

Le séparateur Centrax a été concu spécialement pour la séparation des solides a partir des mélanges des hydrocarbures tres visqueux. Les traits essentiales du prototypye sont:

- les parties de l'appareil qui viennent en contact avec les hydrocarbures sont écartes des éléments d'entrainement méchanique,
- la séparation des solides est éffectuee indépendement de leur characteristiques physiques,
- le besoign du transport des solides inter-étapes dans un systéme au configuration en plusieurs étapes fut eliminé.

Le programme expérimental révéla que la performance d'operation de ce premier prototype fut exceptionnélle en ce que concérne conformance aux speciffications. Lappareil opera sans problémes a la vitése de 4600 rotations a la minute et température de 180°C. Les essais furent arretés par faulte de finir l'alimentation et non par difficultées d'opération. Durant le programme expérimental ont a apporté des améliorations aux instruments de control, au control de température et a l'operation du roulement de base du séparateur Centrax. Des changements temporaires ont etes portes al'ensemble de ce roulement. Ce roulement faudra etre redessine pour les unités qui soit eventuélment construites.

Processing Performance was measured based on the following objectives:

- Quantitative:
  - Separation capabilities of the Centrax under different conditions as evaluated by:
    - clarity of separated liquid,
    - compaction of separated solids.
- Qualitative:
  - Processing or transport capabilities of the Centrax with regard to:
    - feed into the unit,
    - separated liquid exiting by gravity flow,
    - separated solids, cake removal by positive ejection from the separation zone into the receiving section of the separator.

Quantitative measurements showed that the ash concentration of the separated liquid can be reduced to very low levels depending on the feed rate, applied G force, and the composition of the feed. However, separation of identical feed material on the pilot plant scale (Centrax) and on the bench scale showed that ash levels in the separated liquid were consistently higher by a factor of 2 to 5 for the former. For example, ash concentration of separation liquid was:

- Centrax 0.17 %
- Bench Scale 0.05 %

This needs to be investigated further.

Preliminary data suggests that some of the separated solids are pushed into the exit lines of the separated liquid during removal of these solids from the separator. This "cross-over" of separated solids back into the separated liquid needs to be minimized or eliminate in subsequent designs. This problem is a strong function of the compressibility or plasticity characteristics of the solids cake, and this problem is at its, maximum for this application. The cake has the consistency of a very viscous liquid at the processing temperature.

La Pérformance D'opération fut mesures a base des critéres suivantes:

- Quantitative:
  - capacité de séparation aux conditions différentes evaluée par:
    - claritée du liquide separé
    - tassage des solides separés
- Qualitative:
  - capacité de traitement ou transport concérnant:
    - l'entrée de l'alimentation,
    - la sortie du liquid separé par graviteé
    - le déplacement de la croute des solides separés par éjection positive de la zone de séparation a la séction de réception du séparateur.

Les mésures quantitatives ont montré que la concentration en cendre du liquide separé peut être réduite au niveaux tres base dépendant de la rate d'alimentation, la force appliquée et la composition de l'alimentation. Cependant, des separations des alimentations identiques a l'échelle pilote et au laboratoire ont montré que les conténues en cendre dans les liquides separés été toujours plus élevées par. Par éxemple concentration en cendre fut:

- Centrax 0.17 %
- Laboratoire 0.05 %

Cet aspect doigt être encore investigué.

Les résultats préliminaires suggérent que une partie des solides séparés sont poussé dans les lignes de sortie du liquide separ durant l'évacuation des solides. Ce "croisement" des solides separés reculant dans le liquide separé devra être élimine ou au moins minimisé au future. Ce probléme est lié diréctement aux propriéte de comprésibilité ou plasticité de la croute de solides et il est maximum a la consistence tres viscueux du liquide a la temperature du traitement.

Qualitative observations showed that there was no build-up of liquids and solids in any part of the Centrax of their respective areas. Liquid exited by gravity and solids were pushed out of the bowl as designed. Build-up of solids under the scraper did not occur in line with the design concepts.

Preliminary hydro-treated tests on the Centrax separated liquid showed that separation by this process appears to have no detrimental effects on the upgrading process. More work is required to confirm this observation.

A computer model was developed for a geneic integrated two-stage coal liquefaction facility with inter-stage deashing. This interactive model predicts the before tax rate of return for this plant and allows the testing of the sensitivity of the economics of producing coal liquids to changes in deashing performance, hydrogen generation systems, coal quality and liquefaction properties and severity of second stage upgrading. A wide range of pricing regimes, plant financing and inflation assumptions can be used in the model.

The main purpose of the model was to evaluate to economic benefit associated with the enhanced liquid recovery associated with Centrax Deashing compared to Critical Solvent Deashing (CSD). A Canadian Lignite with 17% ash (MF) giving 92% overall conversion was used in the analysis.

Des observations qualitatives montrérent que il n'y a eut pas des accumulations de liquide ou solides dans auqune partie de l'appareil. Le liquide sorta par gravité et les solides furent propulsés de la facon prévue dans le déssin. Accumulation des solides au desous du grattoir, pourtant n'été pas prevu.

Des essais préliminaires de hydro-traitement des liquides separés par Centrax indiquérent que la séparation des solides par cette mèthode ne presente pas des éffects détrimentales pour l'étape de rafinage. Il faudra continuer ce travail pour confirmer cette observation.

Sandwell Technologies développa un model economique sur son ordinateur pour une usine generique de liquefaction du charbon a deux-étapes avec enlévement des cendres entre les deux étapes. Ce model est interactive et predit le taux de retour de l'invéstissement avant - tax pour cette usine, permétant en même temps de eprouver la sensitivité des critéres économiques dan la production des liquides a partir du charbon en fonction de changements dans la performance de l'unité de enlèvement des cendres, du mode de production du hydrogéne, de la qualitée du charbon et ses proprietés de liquefaction aussi que de la sevéritée de rafinage. Il y a dans ce model la possibilité de varier les regimes de prix, de financement aussi que les hypotéses sur l'inflation sur une grand rangée.

Le plus important but de ce modèle fut de evaluer les bénéfices associés avec la meilleure recupération des liquides due au application du procédé Centrax par comparaison avec le procédé CSD (extraction avec un solvent en état super-critique). Le cas d'une lignite Canadiénne ayant 17% (sec) de cendre, qui donne 92% conversion fut utilisé dans cette analyse economique.

Capital costs for each of seven main plant sections were estimated using cost factors reported in the studies. Since the Centrax Separator is still under development, the cost of this section has the greatest uncertainty. For the purpose of this analysis, a conservative estimate of $9,200 per TPD of feed to the deashing unit operation has been used. This is approximately 2 1/2 times more costly than for the CSD system. Nevertheless, the model predicts that Centrax Deashing improves the return on investment by 2.5 percentage points when the plant is configured using residuals and coal as gasifier feed for hydrogen production. With the more complete recovery of product, the Centrax case creates the opportunity of using natural gas for hydrogen generation by steam reforming. In this case, Centrax Deashing results in an improvement of the return on investment over CSD by 7.9 and 8.6 percentage points for the low and high severity upgrading cases respectively. This is almost a doubling of the plant financial return.

The sensitivity of coal liquefaction economics to the capital cost of Centrax Deashing was also investigated. A further doubling of the cost of Centrax Deashing would still give a financial return over 4 percentage greater than for CSD (14% vs 9.9%). It is concluded that the benefits of Centrax Deashing - greater liquid yields, hydrogen production by steam reforming, no requirement external solvent, are such that , if necessary, larger capital costs could be readily absorbed to achieve better financial returns.

Les couts capitales des chaque des sept séctions de l'usine furent estimés a l'aide des facteurs de cout raportés ailleurs. Parseque le séparateur Centrax est encore en état de développement, le cout de cette section a le plus haut degrée de incertitude. Pour le but de cette analyse, on a utilisé une estimation conservative de $9,200 par tonne d'alimentation vers l'unité de séparation par jour ce qui est deux fois et demi plus cher que pour le système CSD. Cependant, le modèle predit que l'application du procédé Centrax augmente le taux de retour de l'invéstissement par 2.5% dans le cas ou la configuration choisi est celle utilisant charbon et résidue comme alimentation au gazogéne pour produir l'hydrogéné nécesaire. Avec la plus complétte recuperation des produits, le cas employant Centrax donne l'alternative de utiliser le gaz naturel pour fabriquer l'hydrogéne. Dans ce cas, l'application du procede Centrax resulta dans une amelioration du taux de retour sur l'invéstissement comparée au cas CSD de 7.9 et 8.6 % coréspondant aux base et haute severite, ce qui veut dire doubler le retour de l'investissement.

La sensitivité des critéres économiques en fonction du cout capitale de l'unité Centrax fut aussi etudié. Augmentant les couts capitales de l'unité Centrax par 100 poucents aura comme resultat un retour de investissement plus de 4 pourcents plus que le cas employant CSD 14% versus 9.9%. La conclusion de l'analyse économique fut que dû aux ses avantages, notamantent le reudement en liquide plus elévé, la production de l'hydrogené a partir du gaz naturel, l'élimination du solvent extérne, Centrax donnera de meilleurs taux de retour sur l'investissement même si les couts capitales soit plus éléves.

Although, some problems remain to be solved, the step from laboratory batch separation to continuous pilot plant operation has successfully been made. It is estimated that a $900,000 CDN, 12 month program with the proper support can progress the Centrax development from this prototype multi-functional single stage unit to a pilot plant consisting of:

- second generation separators,
- first continuous liquid recovery unit,
- integrated assembly made-up of several separators in series closely coupled to each other and the recovery unit,

ready for extended demonstration runs.

Bien que il y a encore des problémes a résoudre, on a passé avec succéss du laboratoire a l'operation pilote en continuu. On estiment que un programme de 12 mois à un cout de 900,000 dollars Canadiens avec le support convenable suffiront pour avancer Centrax a partir de l'étape présente de prototype multi-fonctionale a l'étape pilote qui consistera de:

- une seconde génération des séparatoires
- première unité de récuperation du liquide en continuu
- un ensemble intégré consistant des plusieurs séparatoires en serie couplés-de-pres ensemble avec une unité de récupération.

1.0 INTRODUCTION

Phase III covers the commissioning and preliminary testing of the multi-functional pilot plant Centrax separator. This separator was specially designed for the separation of solids from viscous hydrocarbon liquids made up of oils, asphaltenes and pre-asphaltenes.

Described in Phase I is the laboratory process development work for separation of such mixtures. That work culminated in the development of a unique process scheme which has been patented. By the scheme separation is affected by the displacement of all liquid hydrocarbons, including the oils, asphaltenes and pre-asphaltenes from the solids by a specially selected process-derived extractant which is recoverable by evaporation and which is soluble with the oils, asphaltenes and pre-asphaltenes throughout all process steps. That is, in the first stage, the insoluble solids are separated from the solution formed by the addition of the extractant by the application of centrifugal forces to produce a clear centrate and a compacted solids phase with minimal residual liquid hydrocarbons.

In subsequent stages the residual asphaltenes and pre-asphaltenes in the interstitial spaces of the compacted solids phase are separated by the further addition of an extractant, followed by centrifugal separation to form a solids phase from which the asphaltenes and pre-asphaltenes have been displaced by the process-derived extractant. The volatile process-derived extractant is recovered by atmospheric flash evaporation in the final stage to yield a dry and friable ash concentrate consisting of ash and unconverted coal.

This particular process scheme allows for the complete separation of such solids from the hydrocarbon liquids without the loss of any of these liquids. Although this accomplishment is essential for economic reasons as discussed in this report, it gives rise to the need for a mechanical centrifugal separator which has unique features and capabilities unavailable in any existing equipment.

Phase II describes the details of the hardware development of a centrifugal separator (prototype 1) with the required capabilities. The essential features of these prototype are that:

- the processing part of the apparatus is in essence isolated from the drive section,
- the unit can separate solids independent of the physical properties of these solids, and
- the requirement of inter-transport of solids in a multi-stage configuration has been eliminated.

This makes possible the separation of relatively small particles (micron size) which are very abrasive, sticky, and nonfilterable very viscous hydrocarbon liquids. In addition, the Prototype unit is capable of removing a centrifugal cake which has the consistency of a stiff "putty".

The mechanical specifies of this prototype are described in the Phase II report. Operating details and preliminary test results are outlined in the following chapters of this report.

The trade name selected for the prototype separator is Centrax TM. Centrax is also used as a generic name to describe the patented separation process, and in addition, is used to denote the whole pilot plant which consists of process vessels, pumps, hydraulic power pack, control panel, instrumentation, and the prototype separator.

2.0 CENTRAX PILOT PLANT

The pilot plant is shown schematically in the Piping and Instrumentation Diagram Figure 2.1. The assembled pilot plant is shown in photographs Figures 2.2 and 2.3. It is located at the research and development facilities of Sandwell Technologies in Mississauga.

Shown on the right of these photographs is the Centrax Separator above the hydraulic power pack while the control panel is located on the left side. Visible in the background are the feed and product vessels, denoted as vessel number 1 through 4 on the P & I diagram, Figure 2.1.

2.1 Installation and Assembly

The Centrax separator was assembled and balanced at Orenda, while the hydralic power pack was bought as a unit from National Hydraulics. All other equipment was assembled by the staff of Sandwell Technologies. They were also responsible for the installation of each of the units.

The floor plan of the pilot plant was laid out in such a way as to allow for installation in a modular manner. The plant can be dis-assembled, and re-installed at a possible testing site, with the minimum of disconnections. The major pieces can be transported as individual modules. In addition, the pilot was laid-out in such a way as to provide for installation on a movable testing facility.

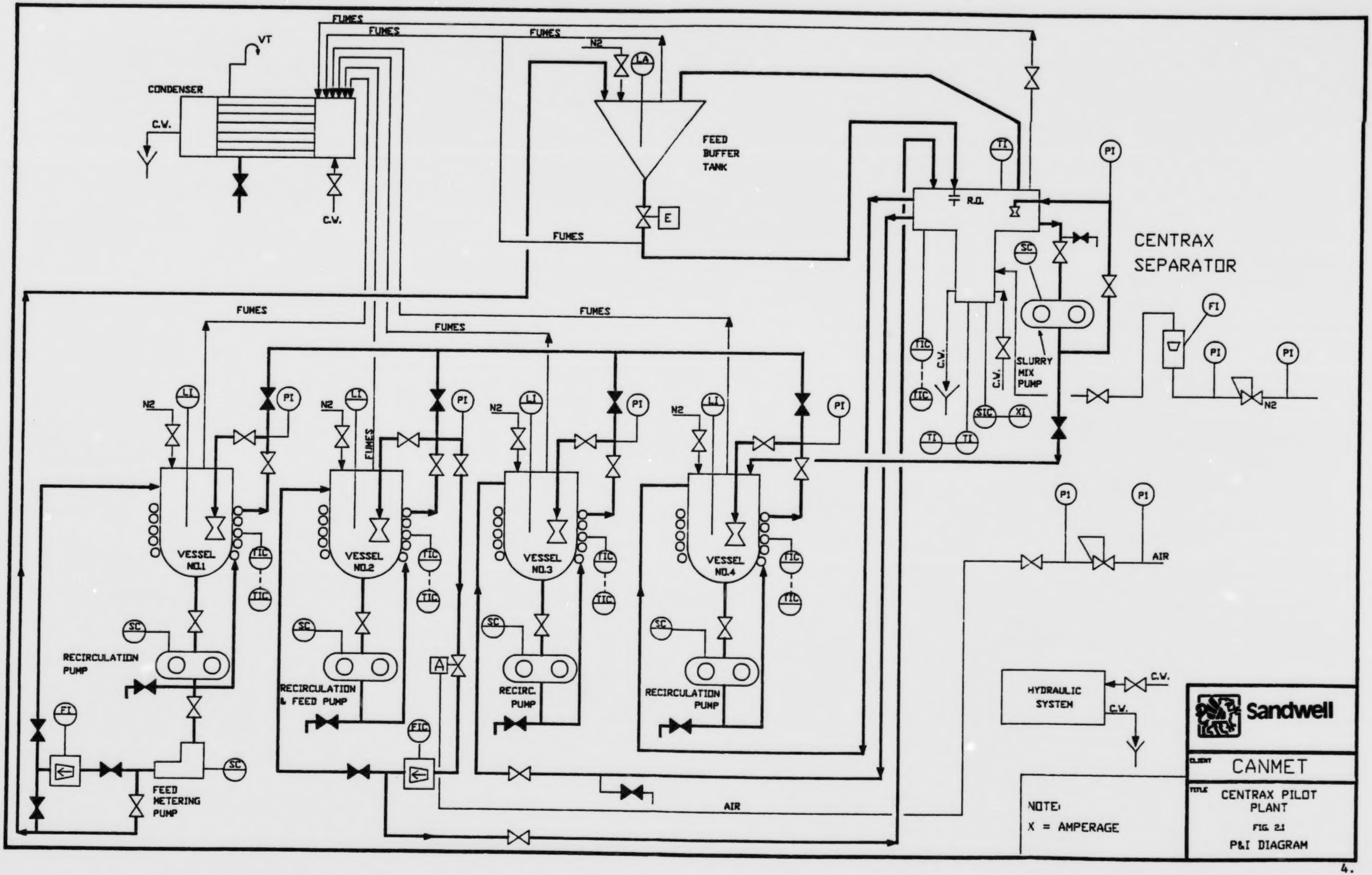
The modular approach and particular layout were adopted with the view of conducting future test work at an other than the Sandwell Technologies location.

2.2 Process Description

The process description is addressed in several sub-parts. First, the process operation of the Centrax separator is discussed, second, the description of a typical operation of the pilot plant as a whole is provided, and third, the control program operation is described.

2.2.1 Operation of the Centrax Separator

The Centrax separator is presented in schematic form in Figure 2.4. It consist of basically of two sections. The top part, which is the processing section, and the bottom part which is the mechanical drive mechanism. Only the top section is heated with heating tape and insulated as shown in Figure 2.5. Heat flow from the top section to the bottom section is minimized by the thermal barrier indicated by the cross hatched plates in Figure 2.4. Any heat which flows through this barrier into the bottom section is disapated by constant flow of hydraulic oil through the hydraulic cylinder and a check valve mounted in the hydraulic piston. The hydraulic oil is in turn cooled by cooling water in the heat exchanger of the hydraulic power pack.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SLURRY
MIX
PUMP
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FEED
METERING
PUMP
PI
PI
AIR
AIR
HYDRAULIC
SYSTEM
C.V.
C.V.

Sandwell

CLIENT CANMET

TITLE CENTRAX PILOT PLANT

FIG. 2.1

P&I DIAGRAM

NOTE:
X = AMPERAGE

FIG. 2.2 FRONTAL VIEW OF CENTRAX PILOT PLANT

FIG. 2.3 SIDE VIEW OF CENTRAX PILOT PLANT

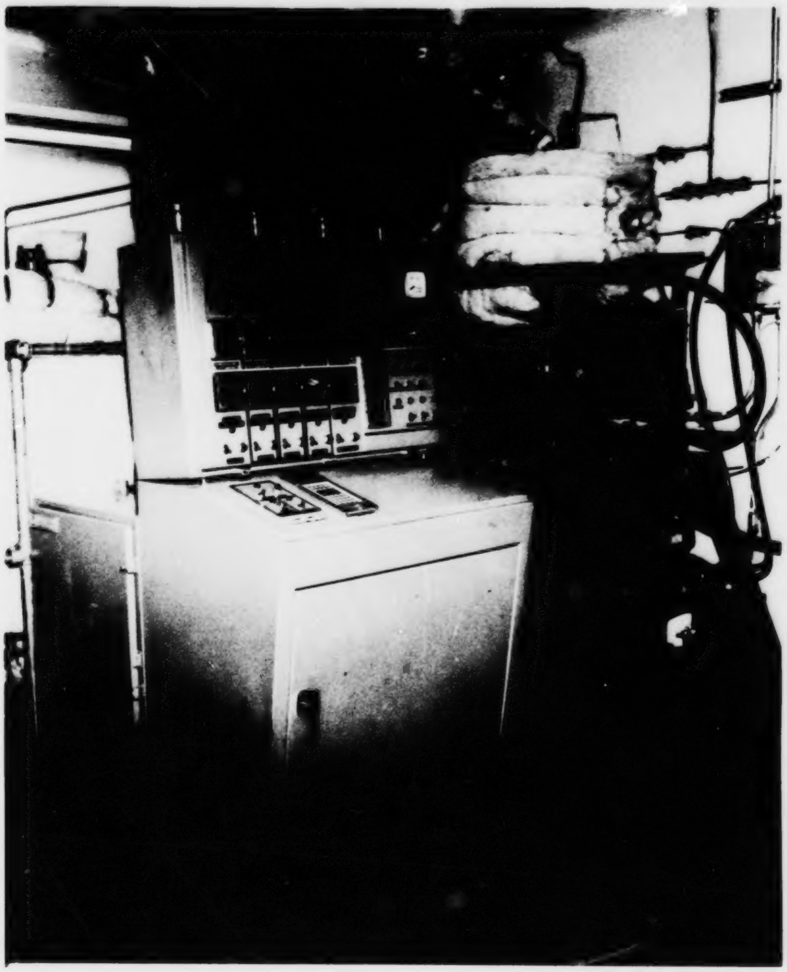


FIG. 2.4 SCHEMATIC OF CENTRAX SEPARATOR

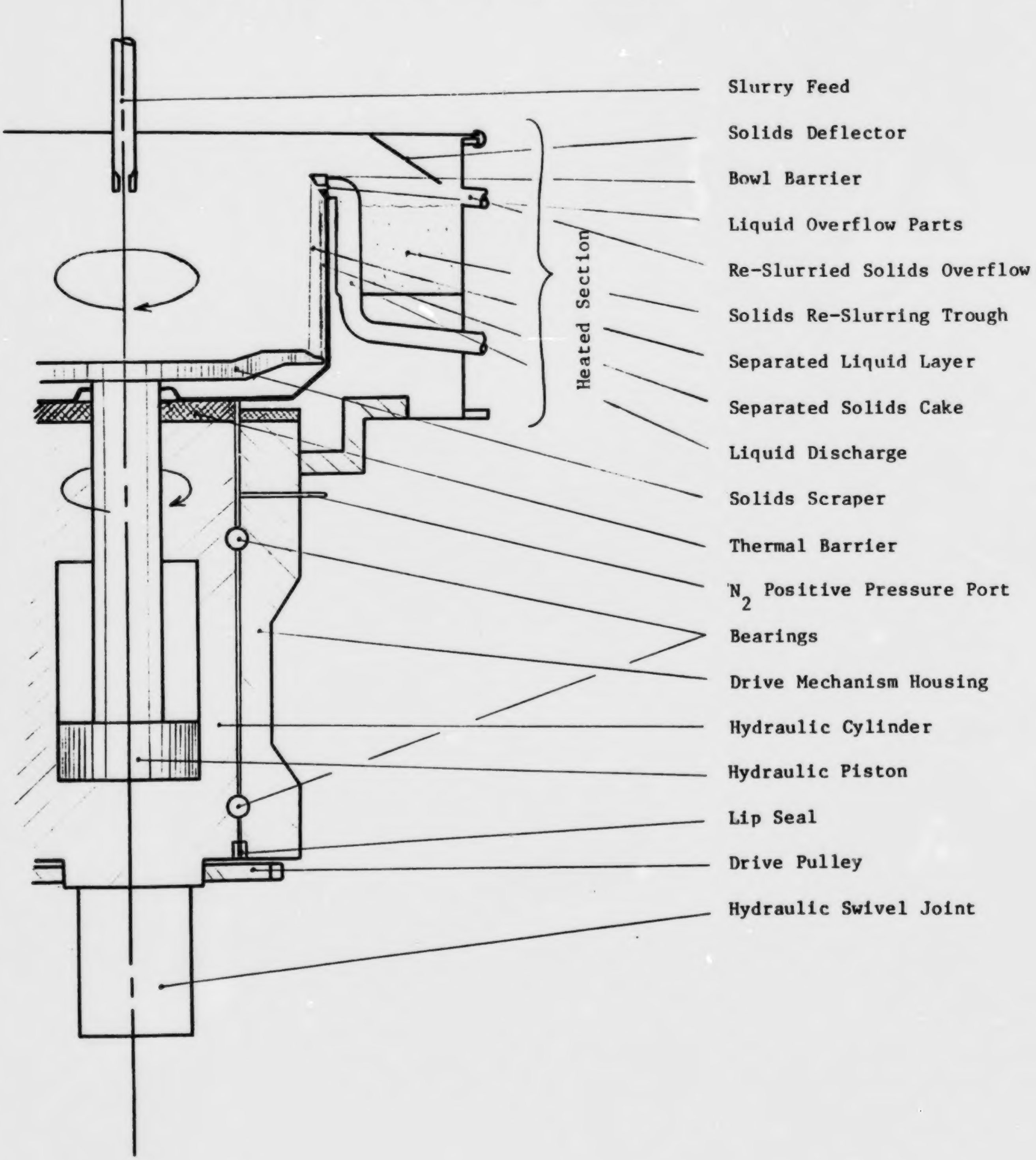
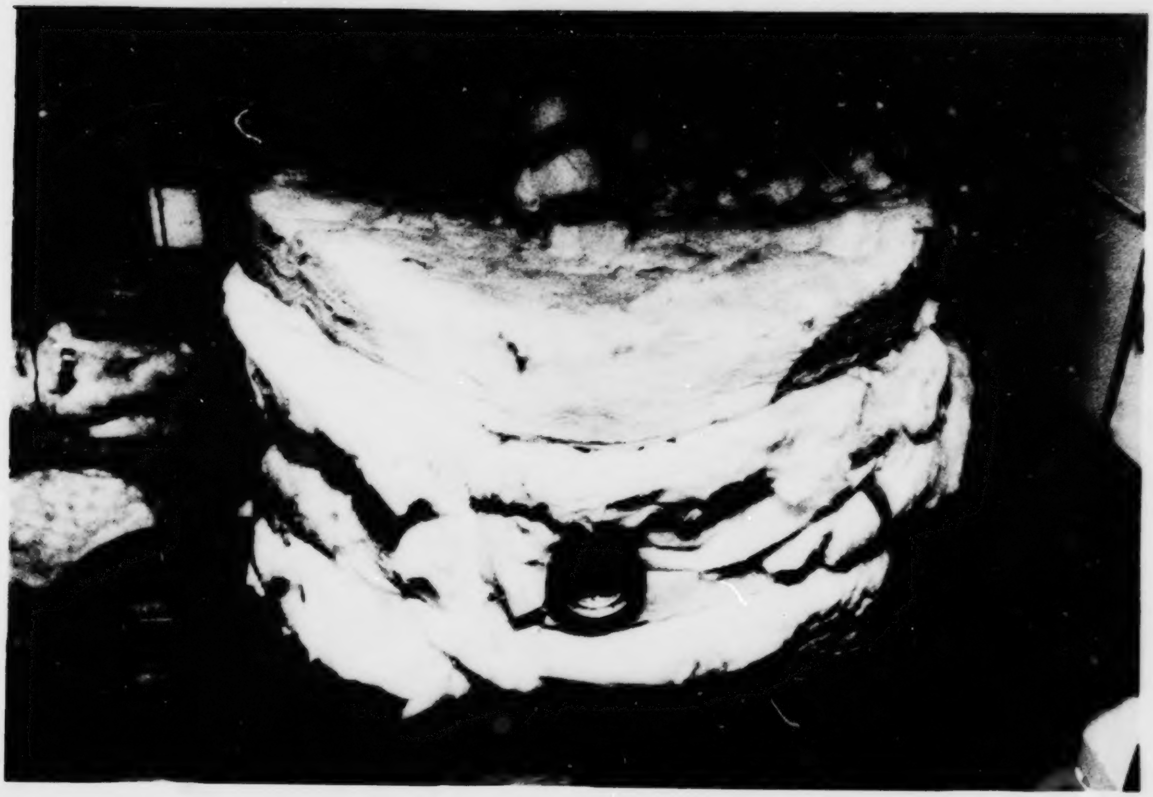

Slurry Feed
Solids Deflector
Bowl Barrier
Liquid Overflow Parts
Re-Slurried Solids Overflow
Solids Re-Slurring Trough
Separated Liquid Layer
Separated Solids Cake
Liquid Discharge
Solids Scraper
Thermal Barrier
N_2 Positive Pressure Port
Bearings
Drive Mechanism Housing
Hydraulic Cylinder
Hydraulic Piston
Lip Seal
Drive Pulley
Hydraulic Swivel Joint
Heated Section

FIG. 2.5 INSULATED TOP SECTION OF CENTRAX SEPARATOR



Pre-heated slurry is fed through the slurry feed pipe and nozzle in a well defined stream directly onto the top of the Solids Scraper. The solids scraper which is, for instance, turning at 4600 RRM, together with the hydraulic cylinder, piston, piston rod, and separator bowl, spins the slurry outwards by centrifugal force against the bowl wall. Separation is affected by the centripetal action of the bowl wall on the slurry feed. Solids, because of their larger specific gravity form a cake on the bowl wall, while liquids are displaced into a layer on top of the cake. With continuing feed both these layers increase in size until the liquid layer has grown sufficiently toward the center of the bowl to be able to escape from the bowl through the liquid overflow ports. From these ports the liquid flows by gravity out of the liquid discharge channel and into a product receiving vessel.

After a pre-determined time the hydraulic mechanism is activated (discussed in one of the following sub-sections) which imparts an upward motion to the solids scraper. It travels upward at a pre-set speed while rotating at the same RPM as the bowl. This provides for a low relative motion of the metal parts of the scraper against the abrasive solids layer on the inside of the bowl. Liquid and solids are pushed upward by this motion, with the liquid exiting through the liquid overflow parts, and the solids forming a "dam" in front of the scraper under the influence of the scraper force and the superimposed centripetal force of the bowl wall. Special design of the liquid overflow ports directs the solids past these ports. At the end of the stroke the scraper pushes the solids out of the bowl wall over the barrier at the top of the bowl.

The solids, after discharge from the bowl, depart from the bowl in a tangential trajectory and impinge on the solids deflector ring. At that point they are directed downward into the solids re-slurrying trough. This trough may either be empty or filled with extractant. In the empty mode the solids are collected in this trough and manually removed after a simulation test. Because of the accessable top design (see Figure 2.5) it will be possible to automate the solids removal operation in an industrial unit. The basics of this design make it possible to remove solids from this trough by positive mechanical action inspite of the tendency of these solids to adhere to solid surfaces.

In the case where the solids are discharged into a trough filled with extractant, the solids transfer is affected by gravity flow of the slurry into either a receiving vessel or into the next stage Centrax separator. This would be part of the operation of a multi-stage set-up which can be simulated in this pilot plant by successive separation, storage, and transfer from receiving vessel to slurry feed vessel. The slurry in this trough is well mixed by recirculating the slurry with a pump through two eductors placed at the bottom of this trough (see Figure 2.4).

For a commercial unit the use of the recirculative pump can be eliminated to reduce the abrasive wear of the metal parts of such pumps by the installation of side entry mixers. The seals of such mixers would of course be placed above the liquid level to minimize wear.

Wear on the bowl wall by the abrasive solids is eliminated by the build-up of a one millimeter thick cake layer on this wall. Instead of designing the scraper with a seal arrangement, it was designed with a deliberate clearance of, in this case, one millimeter. This means that any relative motion is only between the scraper and the solids, and not between the scraper and bowl wall.

The combination of low relative speed between the to be removed solids and the solids removal mechanism, and the above described design feature eliminates, in essense, the excessive wear problem so prevalent in other centrifuges.

The angle of 45° at the bottom of the bowl wall serves to guide any of the solids which end up under the scraper back upward by the centripetal force, and back in front of the scraper. Solids build-up under the scraper is prevented in this way. Liquid will also not accumulate in this area due to this design feature.

Vapours are removed from the separator top section by fume lines shown on the P & I diagram, Figure 2.1. As shown on this figure and Figure 2.4 nitrogen is metered into the drive mechanism part of the separator through the N_2 positive pressure port. Nitrogen is forced to flow upward from the drive mechanism section into the operating section since its flow downward is impeded by the lip seal located at the bottom of the separator (Figure 2.4). The enclosed unit is thus kept under positive pressure in a nitrogen atmosphere. This served two purposes, one is that the hydrocarbon components were blanketed with nitrogen and second it prevented the entrance of hydrocarbon vapours into the drive mechanism part. The first point eliminates the hazards of formation of potential explosive hydrocarbon - oxygen vapour mixtures, and the second eliminates the problem of hydrocarbon vapours condensing and accumulating in parts of the drive mechanism. This would be of special concern as a potential cause of damage to bearings and seals.

Contamination of seals and bearings, specially at high temperatures, with process materials is a potential problem with other centrifuges. In this case the process materials consist of hydrocarbon vapours/condensate and possibly entrained submicron ash particles. Such a mixture might cause excessive wear due to its poor lubricating properties, and second it might destroy certain seals because of its excellent solvent properties. These potential problems, which lead to poor reliability and high maintenance costs, are eliminated from the Centrax separator because of its unique design.

Since all rotating parts of the Centrax are interconnected, it can be shown by a force balance that the bearings are only subject to radial loads and not thrust loads. Depending on the friction co-effecient of the solids cake material such a thrust load might be quite considerable and lead to a very difficult bearing design problems, specially at high speeds. All thrust forces for this unit cancel each other which made possible the use of ordinary bearings lubricated with grease. Also, it minimizes any limitations with regard to progression into separation requirements which require a relatively large G force.

These special design features of the Centrax provides thus for the following:

PROCESS

- Handles non-filterable solids
  Discharges solids directly into a transfer medium to allow for:
  - relatively trouble-free transfer of difficult materials
  - complete redispersion of solids for effective extraction

- Unit is completely enclosed which allows for operation in, for instance, an inert gas environment and/or contain harmful or noxious vapours

MECHANICAL

- No thrust load on bearings
- wear on bowl and scraper by solids discharge is limited to scraper wear ring
- Wear on scraper wear ring is minimized by the application of a relatively low speed differential between the ring and the solids
- Operating environment is essentially isolated from the drive mechanism with regard to transfer of mass:
  - solids
  - liquids
  - vapours
- Unit has the capability of operating at relatively high temperatures without unduly overheating the drive mechanism by means of:
  - minimizing the flow of heat betwen the operating environment and the drive mechanism
  - built-in heat sink, which transfers the 'leaked' energy from the operating environment and the energy generated by the mechanical drive system to a water-cooled heat exchanger
- Design provides for free access to the operating part of the unit to facilitate inspection, cleaning, and maintenance when required
- Operating mechanical features can be altered to suit specific separation requiements without changing the drive mechanism within limitations
- Unit has been designed to allow for addition of additional units with the minimum of interstage transfer equipment to allow for efficient multi-stage extraction/separation
- Special emphasis was placed during the development phase on the objective of accomplishing the process requirements by the simplest mechanical ways to achieve high mechanical reliability.

2.2.2 Operation of Centrax Pilot Plant

For a particular test run feed slurry was either pumped or transferred as broken up solid fragments into vessel number one (Figure 2.1), and extractant liquid was pumped into vessel number two. After introduction of the feed material all vessels and interconnecting piping were brought up to operating temperature. This was usually 180°C.

As soon as the feed material has softened to make it pumpable it was recirculated to enhance the heating rate and prevent settling of solids. Extractant was also recirculated during the heat up period to prevent local overheating and increase the heating rate.

Before turning on the heaters, all equipment was blanketed with N_2 which remained in effect until the equipment had cooled after completion of a run. Also, at this time, cooling water was introduced to the condenser to prevent the emission of hydrocarbon vapours into the environment.

When the feed material and equipment were up to operating temperature all equipment was readied for a test run. This involved the setting of the flow rates of the feed and extractant, start-up of the recirculation pumps of vessels number 3 and 4, and start up of the Centrax separator and hydraulic system. Feed and extractant flow was started after the Centrax reached operating speed. It was brought up to speed in the order of less than a minute.

A run lasted usually from half an hour to one hour depending on the amount of feed material being processed and on the feed rate. During the run data was recorded on log sheets (presented in Appendix 1) and samples taken for analysis after the run. The separated liquid was collected in vessel number three. It was recirculated and kept at operating temperature to prevent solidification and allow for sampling at the end of the run, and for pumping to the approximate storage container. The separated solids were either collected in the solids re-slurrying trough of the Centrax separator, or they were re-mixed with the extractant in this trough from which the slurry overflowed into vessel number four. This material was also recirculated for sampling and transfer at the end of the run. When the separated solids were introduced into an extractant the run was started with a heel of extractant in the Centrax trough to which was added additional extractant at a pre-determined rate throughout the run. The contents of this trough were pumped with the slurry mix pump (Figure 2.1) into vessel number four with the overflowed amount at the end of the run.

Extractant and Slurry Feed Loops

The extractant was metered into the solids re-slurrying trough of the Centrax separator during any particular run. Flow was controlled by means of a pneumatically operated flow control valve which received a feed back signal from a turbine flow meter via the panel mounted programmable flow controller.

The recirculation pump at the base of vessel number 2 supplied extractant to this flow loop with the rest being recycled back through the eductor into the same vessel. This flow split was set manually with the aid of a local pressure gauge (Figure 2.1). Although there was considerable slip through these pumps, which are "rotor gear pumps", they functioned, in essense, as positive displacement pumps. Care needed to be exercised not to "dead head" these pumps during operation.

Feed slurry was fed to the Centrax separator via the Feed Buffer Tank. Control valves were not used in this feed loop since it is felt that such a flow control system of forcing the slurry through a very small orifice would impart excessive shear to the solids. It is hypothesized that such action would negate any positive effect on the separation efficiency as a result of possible agglomeration of the particles. Also, the use of a turbine flow meter might have the same negative action since it contains small inlet and outlet orifices. In addition, such flow meters have a tendency of plugging with a slurry type of feed as apposed to a feed of clear liquor.

For these reasons, it was decided to feed the to be separated slurry by means of a piston feed pump directly from the pressure side of the recirculation pump of vessel number one through tubing and the minimum number of ball valves. The bore of these ball valves was approximately equal to the I.D. of the tubing. A by-pass loop with a turbine flow meter back to vessel number one was used for pre-run flow calibration.

The feed metering pump reciprocated at ~1000 strokes per minute for a typical feed of one litre per minute to provide an approximate continuous flow to the feed buffer tank. Any pulsations were damped in this vessel and by the subsequent flow trough the orifice located at the bottom of the slurry feed pipe. This pipe connects the feed buffer tank and the Centrax separator (Figure 2.1 and 2.7).

The slurry feed loop is shown in Figures 2.6 and 2.7. It was designed with the objectives of: minimizing any particle break-up by attrition, to not provide any areas of solids accumulation, and to convert a continuous feed into a semi-continuous feed as handled by the Centrax separator. The first two points were achieved by proper layout of tubing/piping, while the latter was accomplished with the utilization of a buffer tank.

Flow into this tank is continuous, while flow out is controlled by an electrically operated ball shut-off valve. In the closed position slurry accumulates in the buffer tank until the valve is opened, at which point the feed is re-initiated into the bowl of the Centrax separator. Control of the valve is linked to the position cycle of the solids scraper. This is discussed in detail in a subsequent sub-section of this chapter.

FIG. 2.6 SIDE AND BACK VIEW OF CENTRAX PILOT PLANT

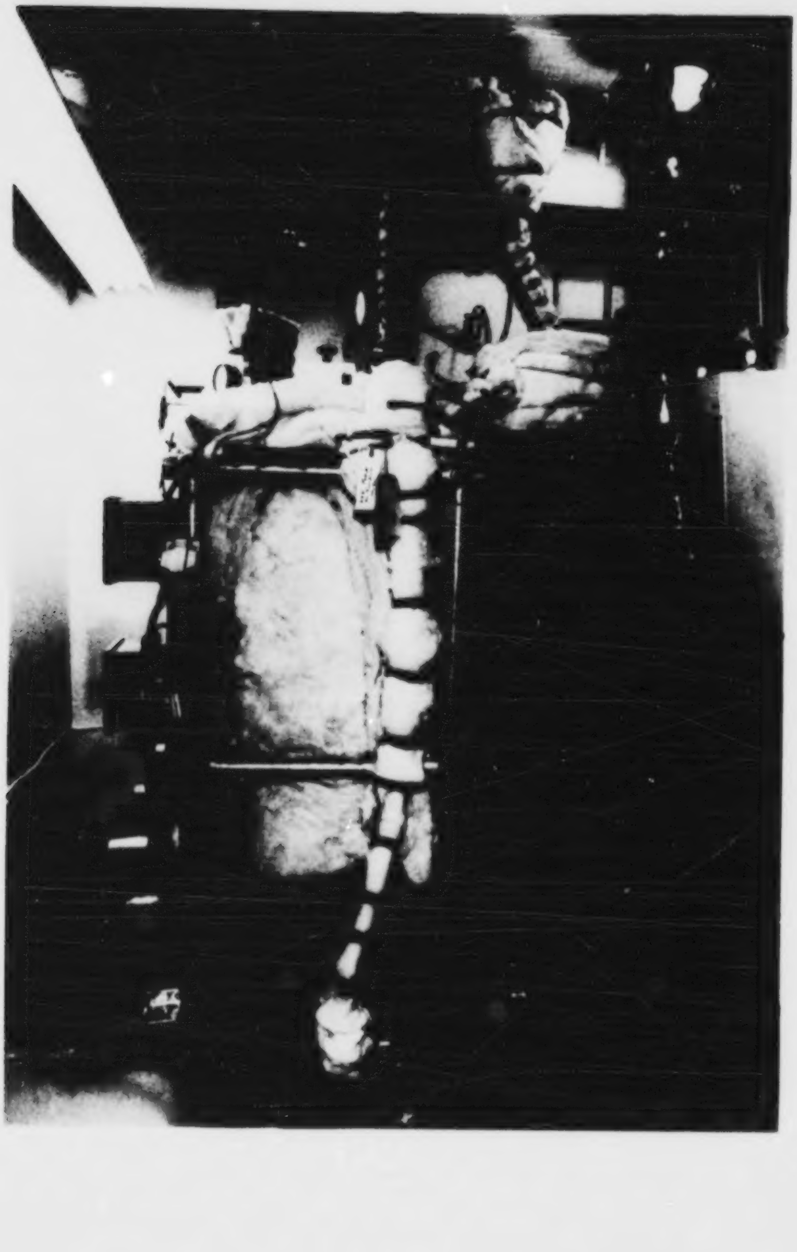
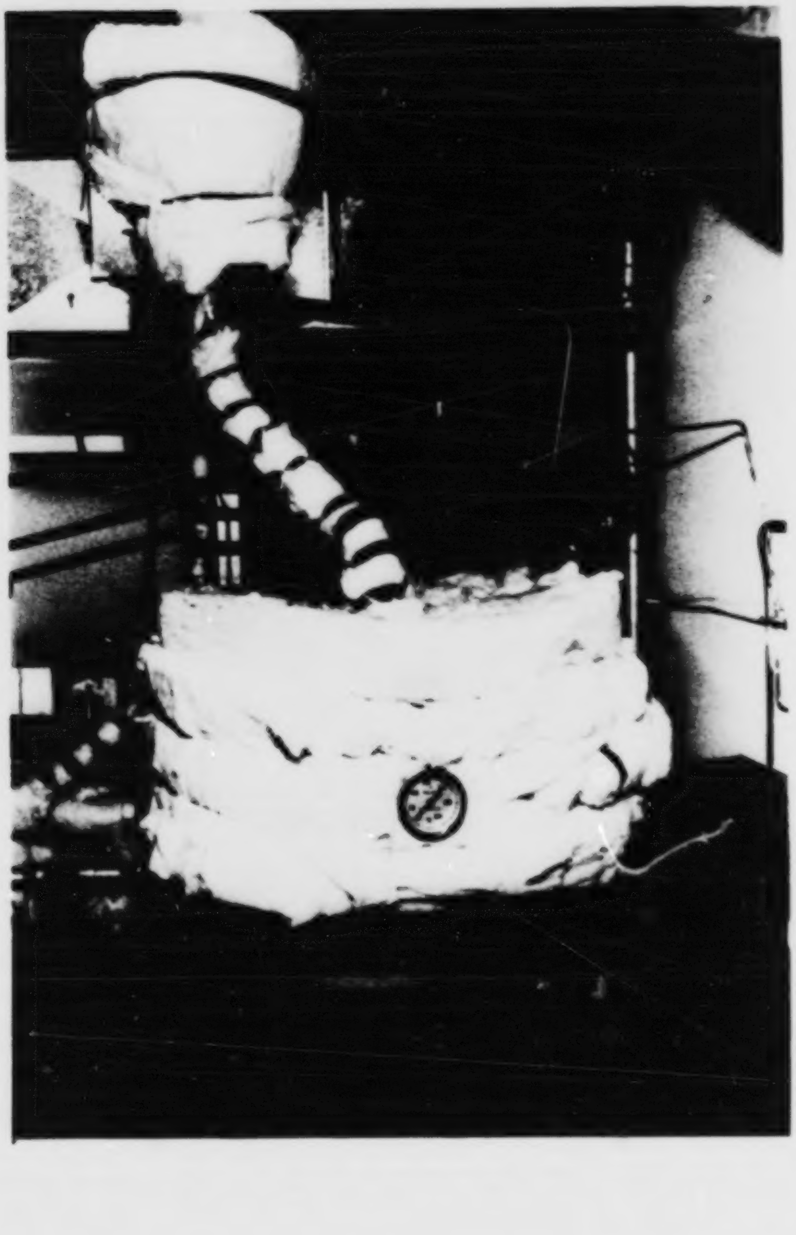


FIG. 2.7 GRAVITY FEED INTO CENTRAX



Mixing And Pumping of Slurries

Except for the feed metering pump, all other pumps were rotor gear pumps. These pumps provide positive displacement of the slurries with minimal attrition and wear. They are suited for high temperature operation and processing of hydrocarbon type of slurries. They functioned as transfer pumps, as recirculation pumps for mixing and for enhancement of heating by forcing the slurry through heating coils. Appendix 4 lists the mechanical details of these pumps.

Mixing is provided in the four vessels and in the solids re-slurrying trough of the Centrax separator by eductors. They are positioned off center and a few centimeters from the dished bottoms of the vessel so as to direct the flow along the bottom of the vessels and upward. This flow pattern, in combination with the location of the recirculative pumps inlets, minimises any dead spots while providing total and gentle mixing. In addition, it prevents local overheating .

The use of eductors has eliminated the problem of seals around stirrer shafts. Although they have been replaced with pump seals for this particular pilot plant. These pumps were required for other than mixing purposes in this pilot plant and thus did not add to the complexity of mechanical equipment. The set-up of a commercial Centrax separator will definitely be different in that it does not require the flexibility of operation as required for this pilot plant which will result in the minimum use of pumps for transfer of abrasive slurries.

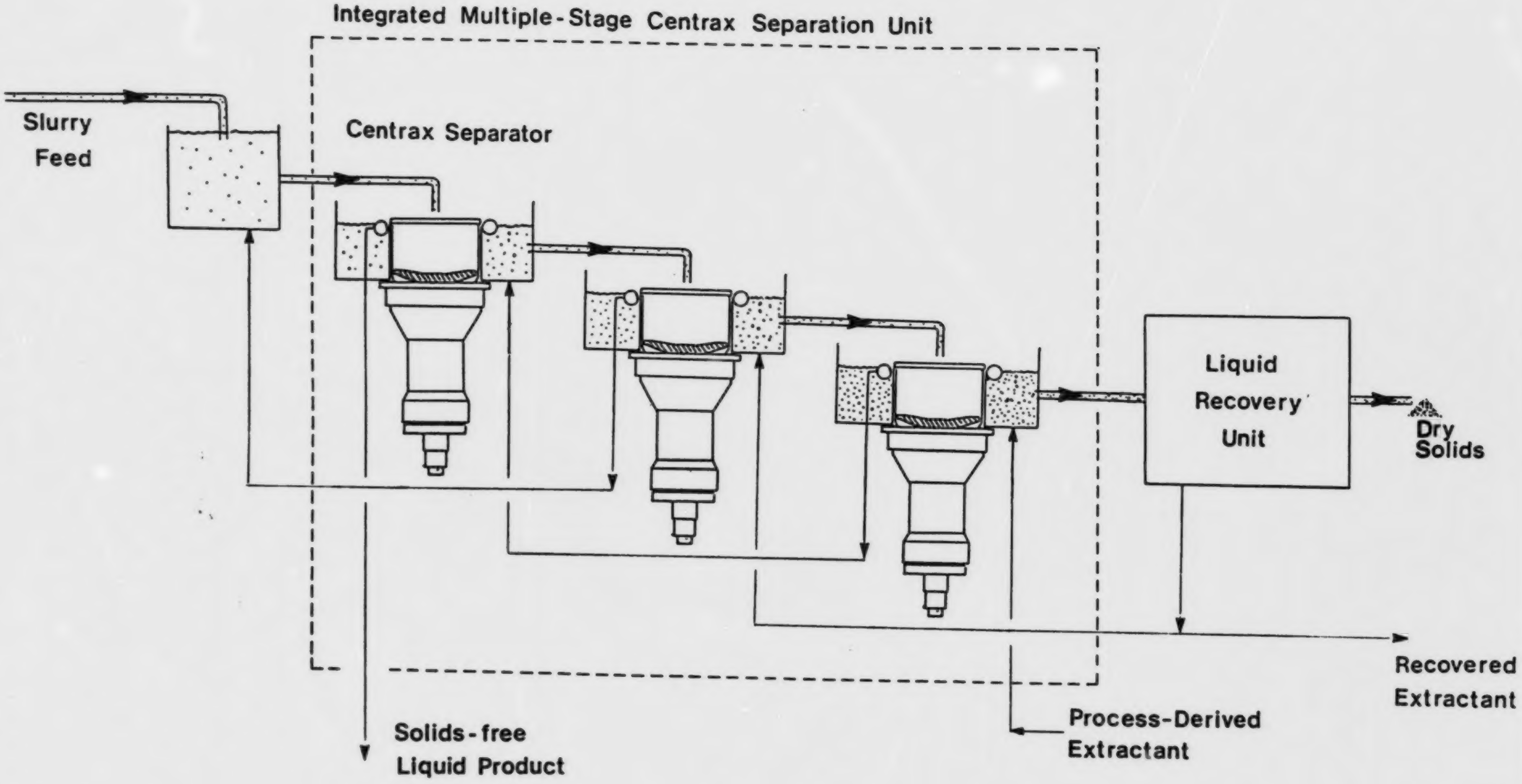
One of the design criteria of the Centrax separator is to minimize contact of slurry material with moving mechanical parts. As shown in Figure 2.8 interstage pumping of slurries has been eliminated in a typical multi-stage set-up. Slurry flows from stage to stage by gravity while liquid extractant is pumped upward between each stage. This should result in minimum maintenance costs and operating difficulties associated with interstage transfer of process flows.

Heating of Process Vessels and Lines

All process piping and tubing were heat traced with thermocouple controlled hot foil heating tape. The feed buffer tank and the heated section of the Centrax separator were also heated with this heating tape and temperature controlled with thermocouples. Heat losses were minimized by insulating all heat traced sections with high temperature fibreglass insulation.

Any sections of either the equipment or piping which dropped below approximately 125°C caused solidification of the type of feed used for this test work. Care had to be exercised to eliminate all "cold spots" and prevent plugging or accumulation of solidified liquid in the system. The function of the installed heating tape /thermocouple / fibreglass insulation was to keep the process material at the specified operating temperature after it was fed from the feed vessels through the Centrax separator into the product receiving vessels number three and four (Figure 2.1).

Fig 2·8 CENTRAX DEASHING PROCESS – 3 Stage Counter-current Arrangement


Integrated Multiple-Stage Centrax Separation Unit
Slurry Feed
Centrax Separator
Liquid Recovery Unit
Dry Solids
Recovered Extractant
Process-Derived Extractant
Solids-free Liquid Product

The feed slurry and extractant were heated up in vessels number one and two respectively, while in vessels number three and four the separated materials were kept at temperature. Wrapped around the side of each of these totally enclosed 110 L vessels was a forty feet length of 5/8 inch stainless steel tubing which served as heat exchanger and insulator to the vessel contents. The tubing was imbedded and covered with heat conduction cement. Energy was supplied to this by means of an electric heating mantle which was in turn wrapped around the outside wall of the cement. All of this and other non heated sections of the vessels were insulated with high temperature fibre-glass insulation.

Temperature of these "jackets" was controlled by two interlocked thermocouples. One was placed under the heating mantle while the other was located in the recirculation pump suction pipe - bottom of vessel section. This allowed for utilization of maximum temperature gradients without local overheating of the vessel contents. Heating was, in addition, speeded up by recirculation of the contents through the tubing wrapped around the vessel which provided a large surface area for heat transfer and enhanced the heat transfer coefficient due to turbulent flow through this tubing.

Level Indicators

Levels were measured in the four process vessels and the feed buffer tank with capacitance type of level probes. The former are panel mounted indicators, while the latter is an alarm only.

The alarm served to alert the operators of any problems with the slurry feed from the buffer tank into the Centrax. Problems could be caused by blockage or vapour lock of the interconnecting piping, or be due to valve failure, or be caused by installation of the wrong orifice, or be the result of some other causes.

The indicators on the four vessels provided back up information on the feed rate out of vessels number one and two, and the rate of product flow into vessels three and four. They served as trouble shooting tools, and allowed for accurate calculation of process flows by analysis of level data as a function of time.

Speed and Flow Controls

All pump drives and the drive of the Centrax separator are DC electric motors with electronic speed controllers. Pump speed control gives the flexibility of using low flow rates for feed or temperature maintenance, and for high rates for inter-transfer between vessels and/or for improving the heating rate during the heat-up period. Also, a low recirculation rate after reaching temperature might aid in agglomeration or at least minimize attribution of the to be separated particles.

The speed controller of the Centrax drive served the dual requirement of being able to vary the G force and provide adjustable braking action. This latter feature is a definite safety related need, specially for an experimental unit. Bearing deterioration or failure would require that the unit be brought to a quick stop to prevent possible extensive equipment damage. This could be automated by installation of vibration sensors. Such measures were judged to be unnecessary for this pilot plant since it was under constant operator surveillance during test runs.

Power Consumption of Centrax Separator

Power requirements for starting and keeping the Centrax separator at operating speed were measured with a panel mounted amperage meter. Additional current requirements for accelerating the slurry feed were superimposed on this reading. Operation with and without feed allowed the determination of mechanical drag and of process related power consumption.

In addition, this measurement served as a significant trouble shooting device. Any unusual increase in current to the electric motor would provide a warning of potential problems in the rotating part of the unit.

Power requirements for pushing the solids scraper up and down can be caluclated from hydraulic flow and pressure measurements. Cycle and axial speed control of the scraper and its interaction with the feed control from the buffer tank is discussed in the next sub-section.

2.2.3 Operation and Control of Solids Scraper

Solids are automatically discharged from the Centrax separator by the solids scraper. Automatic discharge of liquids poses no problems since, as in most nonfiltering centrifugal separators, it overflows over a certain barrier after the liquid level is build-up to a specified depth. All of the difficulty of operation is related to the automatic discharge of the solids. This is accomplished in a number of ways in different devices, from continuous augurs, intermittent knifes/blades, to intermittent pushers. Operation of the Centrax is unique, not in that it uses a pusher, but in its use of a pusher/scraper which positively ejects solids over the full depth of the bowl in combination with the bowl barrier. These design features set it aside from all other units and allow it to process solids which cannot be handled by other units.

Although, slurry feed to the feed buffer tank is continuous, solids are discharged from the level intermittently at full operating speed. Frequency of discharge depends on the solids concentration in the feed. This unit can handle solid concentrations from the ppm range to those just pumpable slurries as explained below.

Solids are discharged after build-up of a certain cake thickness on the inside of the separator bowl. The thickness of this layer is estimated for this pilot plant operation by calculation of solids deposit rate. This can be automated by either measuring the solids concentration in the slurry feed with a signal transmission to a microprocessor which interacts with the control of the scraper, or by means of a non contact measurement of the actual thickness of the deposited cake. The open/accessable top design of the Centrax would make this possible.

The scraper can either be operated in the automatic mode or be controlled manually with override controls. Figure 2.9 is a photograph of the hydraulic power pack used to raise and lower the scraper. A cycle consists of:

1. Holding the scraper in the bottom position for a pre-programmed time interval, during which the slurry feed control valve located underneath the feed buffer tank (Figure 2.1) is held in the open position, and hydraulic oil is pumped through a pressure relief valve located in the hydraulic piston (Figure 2.10). Maintenance of hydraulic flow through the hydraulic cylinder serves as a heat sink for the drive mechanism of the Centrax.

2. Activation of the solenoid operated hydraulic directional valve which connects the bottom of the hydraulic cylinder to the pressure side of the hydraulic pump and the top side to the reservoir of the hydraulic power pack. This moves the scraper upward at a preset speed as controlled by one of the flow control valves shown on schematic Figure 2.10 and photograph Figure 2.11.

3. After reaching the top position the hydraulic oil is directed over a pressure relief valve and momentarily by passes the cylinder. Top and bottom positions are determined by letting the hydraulic piston impact on the top and bottom of the hydraulic cylinder respectively. These positions can be adjusted with shims as shown in the detailed drawings presented in the phase II report. The top and bottom of hydraulic cylinder were designed to provide a slight buffer action.

4. The scraper is held in the top position in the automatic mode for approximately one to two seconds after which the hydraulic directional control valve is shifted to push the hydraulic piston down. The downward speed is controlled by the other hydraulic flow control valve (Figures 2.11 and 2.10). After reaching the bottom of the hydraulic cylinder the cycle is complete. In the manual mode the scraper can be held in the bottom and top position indefinitely. However, the up and down stroke velocities are controlled in the same way as in the automatic mode.

5. In the automatic mode the slurry feed control valve is closed and opened during the upstroke and downstroke of the scraper. It is closed as soon as the scraper starts on its upstroke, and it is opened again at a preset time after the scraper reaches its top position while it is still in its downstroke. This valve must be manually opened and closed from the control panel when operating in the manual mode.

FIG. 2.9 HYDRAULIC POWER PACK

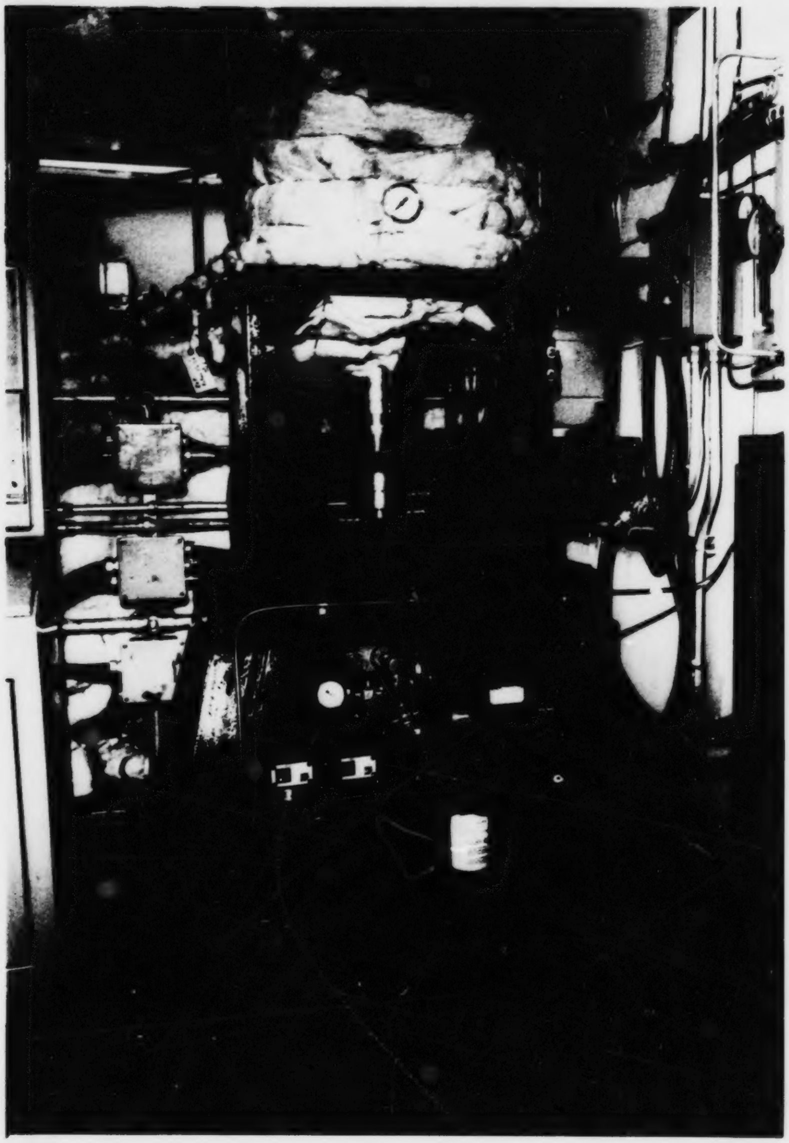


FIG. 2.10 HYDRAULIC SCHEMATIC

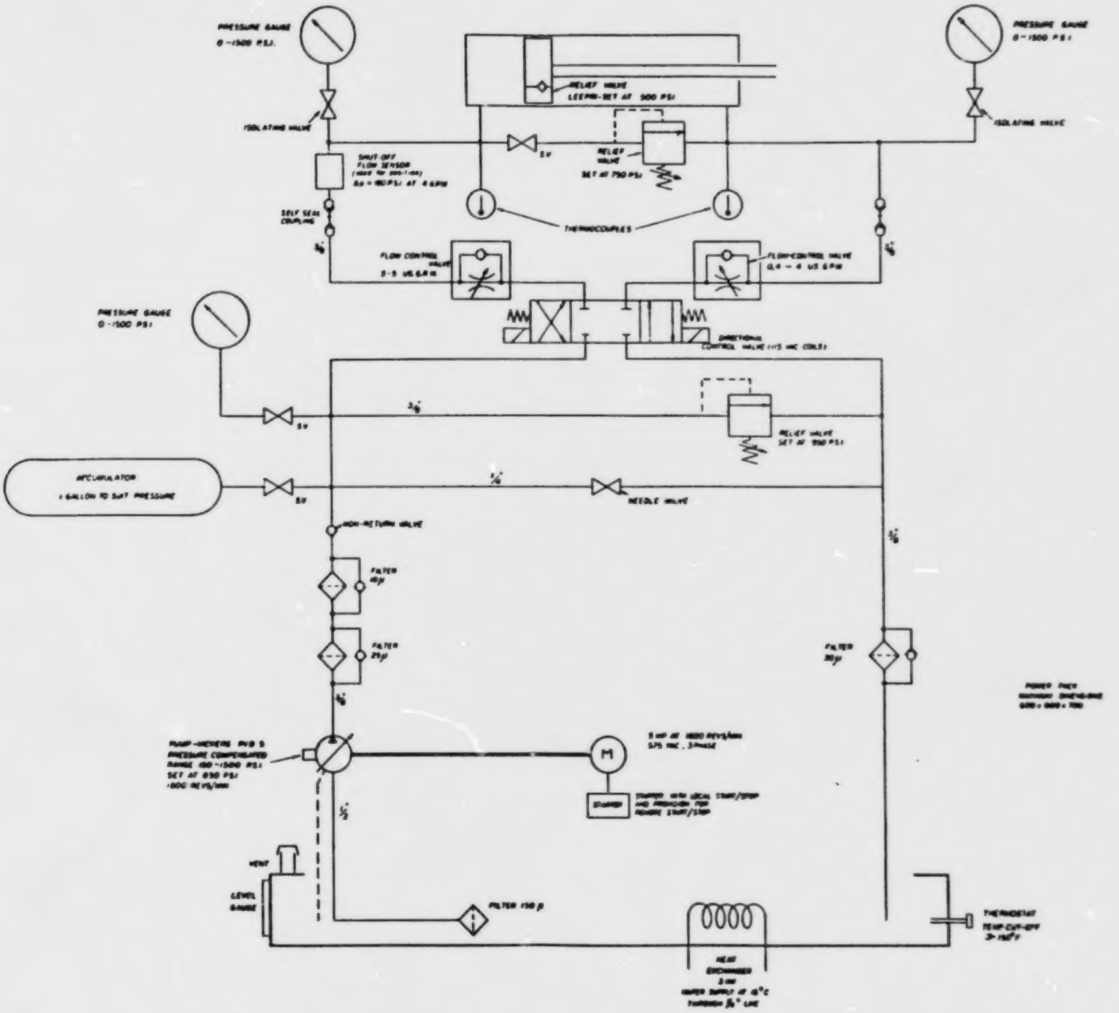
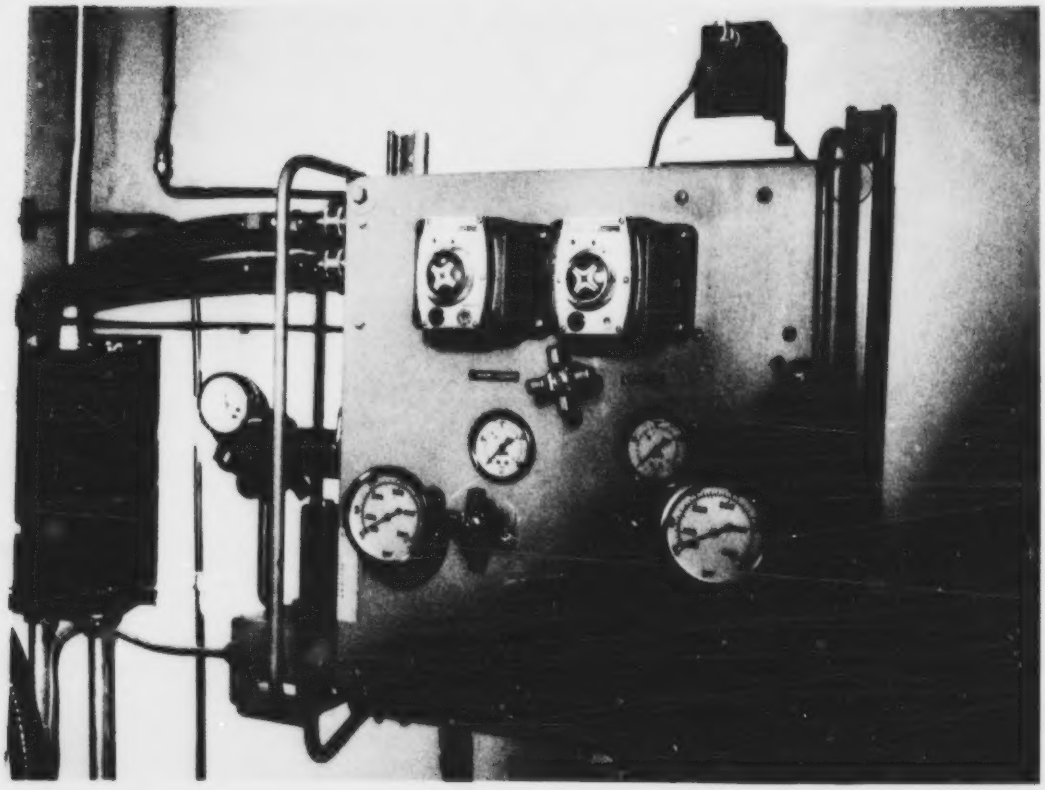

PRESSURE GAUGE 0-1500 PSI.
ISOLATING VALVE
RELIEF VALVE LEEPRI-SET AT 900 PSI
SV
RELIEF VALVE
SET AT 750 PSI
PRESSURE GAUGE 0-1500 PSI
ISOLATING VALVE
SHUT-OFF FLOW SENSOR
SELF SEAL COUPLING
THERMOCOUPLES
3/8"
FLOW CONTROL VALVE
FLOW CONTROL VALVE
DIRECTIONAL CONTROL VALVE
PRESSURE GAUGE 0-1500 PSI
SV
3/8"
RELIEF VALVE SET AT 950 PSI
ACCUMULATOR
1 GALLON TO SUIT PRESSURE
SV
1/4"
NEEDLE VALVE
NON-RETURN VALVE
FILTER
FILTER 25μ
3/8"
FILTER
3/8"
POWER PACK
PUMP
PRESSURE COMPENSATED
SET AT
M
STARTER
1/2"
VENT
LEVEL GAUGE
FILTER 150 μ
HEAT EXCHANGER
THERMOSTAT
TEMP CUT-OFF

FIG. 2.11 EXTERNALLY MOUNTED HYDRAULIC CONTROLS AND INDICATORS



Upstroke speed is set so as to approximately equal the residence time of the pushed out liquid layer to that of the liquid which is fed in and exists with the scraper in the bottom position. This is only an approximation since the liquid velocity profile in the axial direction is probably quite different for the two cases. The accumulated slurry in the feed buffer tank is dropped into the separator bowl when the slurry feed control valve is opened. This fills-up the bowl close to the liquid overflow level, and minimizes the cycle "dead time". That is, the time interval during which no separation is taking place. Again, residence time considerations are the determining factors.

At the heart of the above described cycle is a programmable controller, the P.L.C. shown in Figure 2.12. It activates the solenoids of the hydraulic directional control valve (R1 and R2), and the electrical control of the slurry feed control valve (R3). In addition, it resets and holds the ouput, signal 4 to 20 MA out of the programmable flow controller indicator at 4 MA during the bottom dwell time of the scraper. A programmed timer in the P.L.C. maintains this condition for a pre-set time. Once disengaged the Stauff flow meter, which measure the hydraulic oil flow to the hydraulic cylinder, has its square wave signal transformed to a 4 to 20 MA signal in the flow controller, which in turn sends this signal to the scraper position indicator. This unit is calibrated such that 4 to 12 MA equals the amount of hydraulic oil required for the complete upstroke of the scraper, while 12 to 20 MA corresponds to the amount used for the downstroke.

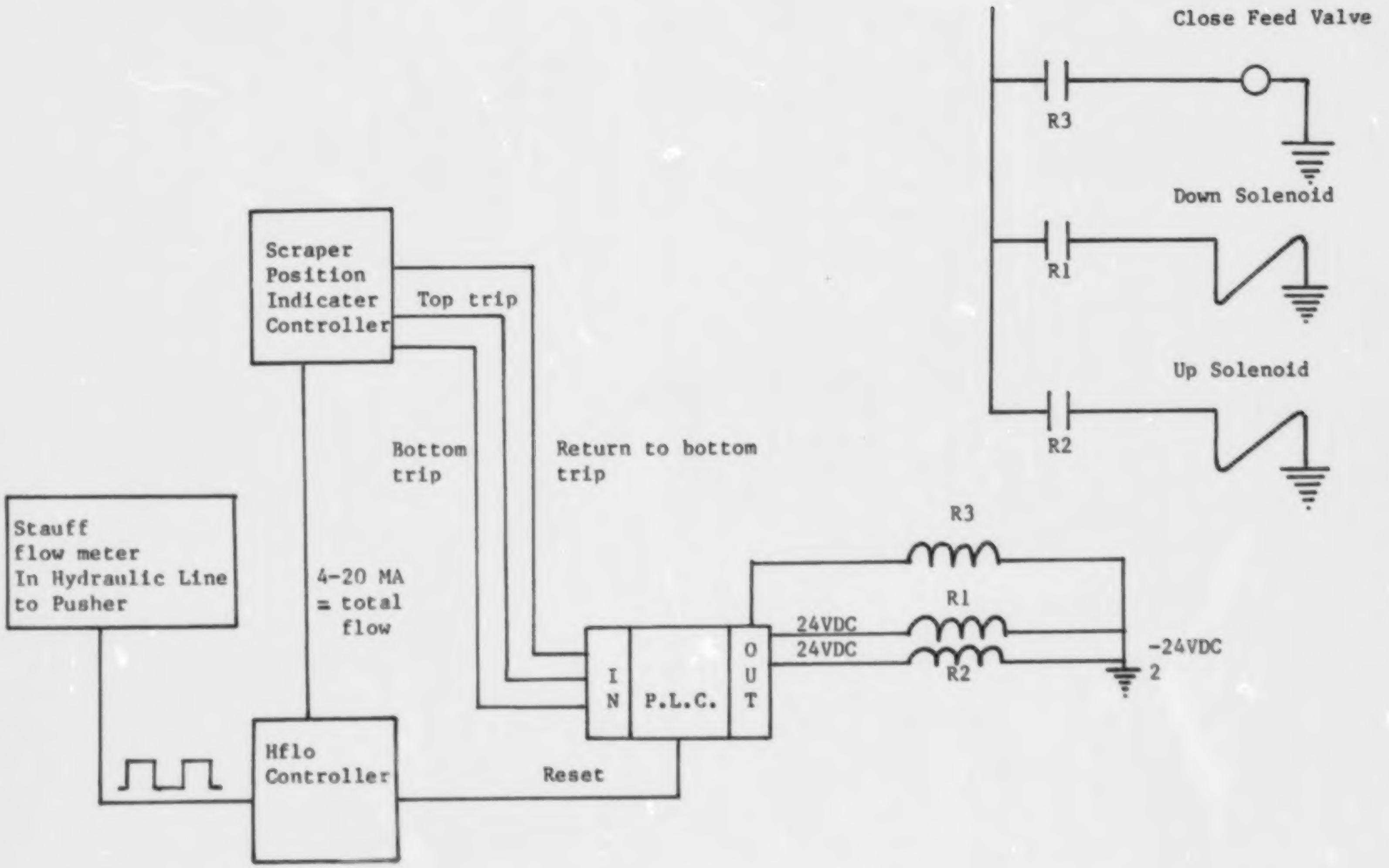
At 12 MA a contact closes in the position indicator which indicates to the P.L.C. that the scraper has reached its top position. The P.L.C. then activates the appropriate solenoids and starts a second timer which holds the slurry feed control valve closed for a pre-set time. At 20 MA an other contact closes in the position indicator which causes the P.L.C. to reset the flow controller to 4MA which in turn sets the position indicator to zero, and at the same time the P.L.C. re-starts its bottom dwell timer to initiate the next process cycle. Interlocks guard against activation of conflicting control modes at the same time.

The position of the scraper, and in turn the position of the hydraulic piston is thus indirectly determined by measurement of the hydraulic flow to the cylinder. This flow measurement is in turn transformed into an electrical signal which then affects control of the process feed flow and of the hydraulic oil flow, completing the control cycle.

Appendix 4 provides equipment details, instrument lists, control panel layout schematics, and a listing of the P.L.C. programming details.

The combination of unique mechanical/process features and the special control arrangement provides for a very versatile unit of which the:

FIG. 2.12 PUSHER CONTROL SCHEMATIC



Close Feed Valve
R3
Down Solenoid
R1
Up Solenoid
R2
Scraper Position Indicater Controller
Top trip
Bottom trip
Return to bottom trip
Stauff flow meter In Hydraulic Line to Pusher
4-20 MA = total flow
R3
R1
24VDC
24VDC
R2
-24VDC
2
I N
P.L.C.
O U T
Hflo Controller
Reset

- Discharge rate of solids can be varied over a wide range, which has as implication:
  - operation of the unit is not fixed with regard to:
    - clarity of overflow
    - dryness of solids cake
  - the same unit can handle:
    - solids of widely different size distributions
    - solids of different relative densities
  - the same unit is capable of:
    - separation of solids from concentrated slurries for bulk solids removal
    - separation of solids from very dilute slurries for clarification to th ppm concentration

Also it:

- operates as a continuous unit operation (although the 'internal' operation is semi-continuous).

To summarize, the capabilities of this unit which distinguishes it over other centrifugal devices are:

- handling of any type of solids
- operation at relatively high temperatures
- essential complete separation of operating environment and drive mechanism
- independent control of liquid and solids residence times to allow for performance optimization of slurries with different separation characteristics
- capability of discharging the solids directly into a transfer extraction medium to provide a mechanical means of high intensity multi-stage solids/liquids extraction.

3.0 COMMISSIONING-TROUBLE SHOOTING/RUN HISTORY

3.1 Commissioning

Upon completion of installation of the Centrax separator, the auxilary equipment (vessels, pumps, etc.), and the control panel with instrumentation, the system was commissioned. It involved the following steps:

1. All equipment was inspected for any contruction debris, and all rotating parts turned by hand to minimize the chance of passing any loose parts of wire or other materials through the pumps and/or Centrax separator.

2. After the mechanical inspection all instrumentation was checked for proper functioning, this included temperature loops, flow loops, speed controls, and scraper control.

3. Special care was exercised with the installation of the hydraulic power pack to make certain that no contaminants entered the hydraulic system. It was thoroughly inspected after which the resevoir was filled with hydraulic oil and the accumulator pressurized with nitrogen.

   Valves were set so as to allow pumping in a closed loop at very low pressure through the pump suction filter (see Figure 2.12), and through the pump and inline pressure and return line filters. Any contaminants in the reservoir, and hydraulic oil were removed by this action. The rest of the system was purged by directing the flow of hydraulic oil through the normal route at very low pressure. Any contaminants picked up in that part of the system were removed in the return line filter.

   After flushing at low pressure for approximately eight hours the pressure relief valves were set at the pressures as shown on Figure 2.12. The system was then operational with the only task remaining of setting the hydraulic flow control valve to get the desired up and down velocity of the solids scraper.

4. Vessel number one was partly filled with water, which was then used as a mechanism for testing the process part of the pilot plant. The water was pumped from vessel to vessel, passed through the flow loops and sub sequently drained. This procedure was repeated a number of times until the water remained clean.

5. As a first step the Centrax separator was as a first step rotated by hand and, as mentioned, thoroughly inspected before turning on the variable speed drive. The bearings had been greased before this, taking care not to "over grease". The unit was then rotated at low speed for a number of hours. During this, it was monitored for vibration, any unusual noise, and for excessive increase of temperature at the bearing locations. This procedure was repeated at increasingly higher speed levels up to the operating speed of 4600 RPM.

Throughout the above tests the scraper and hydraulic piston assembly were in the bottom position. Commissioning of this part of the Centrax separator involved, as a first step, running hydraulic oil through the pressure relief valve located in the hydraulic piston to de-aerate the hydraulic cylinder and hydraulic lines. After this the scraper was moved up and down with the bowl standing still. Again the unit was monitored for any unusual vibrations, sounds, or hydraulic oil leaks. This operation was repeated at different speeds of the up and down movement of the scraper, and lastly the scraper was activated simultaneously while rotating the unit at different RPM's. It was checked for any excessive vibrations by operating the unit with the scraper in the top and intermediate positions as compared to it being at the bottom.

6. After flushing and empty operation of the system, vessel one was again filled with water which was then used as processing fluid. It was heated, pumped, and passed through the Centrax. This provided an initial check on the operation of the instrumentation and on the liquid discharge of the Centrax. Approximate calibrations of instruments were carried out at this stage, realizing any effects due to the difference in physical properties of water and the to be processed hydrocarbon slurries.

7. The next step in the commissioning operation consisted of processing coal liquefaction slurry which was diluted with a relatively large amount of extractant oil. The resultant slurry had a relatively low solidification point and a low relative viscosity. Any cold spots were identified by this operation, and flow problems were made apparent. Instruments were re-calibrated together with the input of control programs written for the P.L.C.

8. As the last step, coal liquefaction slurry with very little extractant was processed at 180^{o}C. This was the most severe test of the complete system. It involved processing at very high temperature with relatively viscous material which had an approximate solidification point of 120^{o}C. Plugging problems and temperature related instrument failures accurred under those conditions.

   This completed the commissioning operation. The next step was to carry-out a number of test runs according to an experimental program. During these runs, additional hardware/instrument and processing problems were noted and subsequently solved. This involved the trouble-shooting part of the project. It is presented in the next sub-section.

3.2 Trouble Shooting/Run History

The majority of runs were completed and carried out according to the pre-run specified conditions. Only one run was aborted before completion. It was the result of processing at too high a feed rate for this particular design.

Listed below in point form are the corrective measures which were implemented throughout the program. They are listed in chronological order, from the start to the end of the work.

References are made to particular parts of the equipment for which either the appendicies or the phase II report need to be consulted. These details are not repeated here to avoid excessive duplication.

Run #1

Before

- Remachined Centrax separator tank and scraper insert to correctly align the liquid overflow ports with the entrance to the liquid discharge,
- added hydraulic flow control valves to achieve proper control over the up and down speed of the scraper,
- took apart and reassembled bottom section of the Centrax drive mechanism to remove excessive "wobble" of hydraulic swivel joint.

Post

- A 3 to 1 gear reducer was installed on the recirculation pump of vessel #2 to increase the pump torgue and make possible operation at low speed,
- the N_2 purge lines into the Centrax separator were modified to improve inside vapour flow patterns,
- to correct leakage of hydraulic oil out of the bottom of the Centrax separator, edges on the hydraulic ports in the pulley assembly were removed so that damage to the O ring seals during assembly was eliminated,
- repaired and re-installed the malfunctioning signal processing card of the turbine process flow transducer,
- re-programmed P.L.C. to monitor reset of the micro-flow controller to eliminate false, "watch-dog" alarms of this controller,
- the type of Loctite used on the screws that fasten the bowl barrier ring to the bowl was changed to a less permanent type to minimize damage to the screws during dis-assembly for inspection purposes.

Run #2

Post

- To correct the problem of excessive noise and heating up of the drive mechanism housing at the location of the lower bearing at speeds above 3500 RPM, disc springs and a 3 mm shim were installed to provide a thrust load on the lower bearing and prevent the balls from "free-wheeling" in this bearing,
- installed a complete set of ROM (read only memory) chips in the micro-flow controller to decrease flow data processing time by a factor of three and give a smoother read-out of the scraper position indicator,

- installed a new CPU (central processing unit) card in the micro-flow controller to solve the problem of the false "watch-dog" timer alarms in this unit,

- replaced the signal converter of the still malfunctioning turbine process flow meter,

- installed a 3500 W heating tape on the outside of the Centrax separator tank, and a thermometer was placed in the solids re-slurrying trough to better control and monitor the temperature of the heated section of the Centrax separator.

Run #3

Post

- The repeated malfunctioning micro-flow controller was replaced with a new unit,

- to further improve the temperature control of the heated section of the Centrax separator:

    - a thermocouple was instralled in the bottom part of the Centrax separator tank which gave a more accurate and faster temperature measurement, and in addition,

    - four 120W cartridge heaters were installed and imbedded in heat conducting cement at the bottom part of this tank,

- the N_2 purge into the Centrax was removed from the common N_2 purge system, and replaced with an independent N_2 supply with a separate regulator and flow meter to provide positive control of the amount of N_2 entering the Centrax unit,

- since the use of the 3 to 1 gear reducer, installed after run #1, on the recirculation pump of vessel number two worked very well for pumping very viscous material, two additional gear reducers were ordered and installed on the pumps of vessels number three and four,

- to correct the problem of the persistent malfunctioning turbine process flow meters, their signal output cards were redesigned and installed,

- application of a thrust load on the outer race of the bottom bearing resulted in a marked improvement in the running of the Centrax at higher speeds, however it was felt that further gains could be made by the redesign and installation of improved mechanical parts to provide a more uniformly distributed thrust load on the bearing; the 3 mm shim and disc springs were replaced by six coil springs placed equal distances apart in a circle and seated in holes in the pulley assembly.

Run #4

Post

- Since the turbine process flow meters were still not functioning without problems they were reconnected to their supports such that the housing with turbine could be kept hot and at the same time keep the electronic part of the meter relatively cool. A too cold housing caused plugging and too hot environment for the electronics resulted in signal transmission failures,

- grease lines to the bearings were lengthened to allow greasing during operation,

- it was found that the coil springs installed, after run #3, were bent in the direction of rotation of the Centrax separator. This strongly suggested that the noise and excessive heating problem at higher speeds of this bearing was indeed due to the suspected problem of the outer race of this bearing not being held in place by the press-fit in the lower bearing housing of the drive mechanism housing. Although, it was recognized that this section of the Centrax needs to be redesigned, as a temporary measure, the approach of holding the outer race in place and have it make firm contact with the balls by the application of a thrust force on it was continued. To prevent the springs from being bent again the exposed spring length was minimised by means of a 2mm spacer ring with holes for the springs to pass through. Additionly the previously used springs were replaced with higher tension springs,

- to improve the data collection during runs 5060-AC-C1 level probes were installed in vessels number three and four in addition to the probes already in place in vessels are and two,

- some of the drain lines and gravity feed lines were replaced to minimise or eliminate any flow problems in them.

- fiberglass insulation used on vessels and lines was changed to AW type 2 crushed as a precaution against fire.

Run #5

Post

- Installed a two piece steel heating band to replace the "burned-out" heating mantle of vessel number two as a temporary measure,

- to improve the ease of operation of the pilot plant and to further improve the accuracy of the process measurements a sonic alarm was added the high level warning system of the feed buffer tank, and a special steel dip-stick was subsequently used in all runs to calibrate the level probes in the four tanks at the operating conditions of each run.

Run #6

Post

- Removed the temporary heating band from vessel number two and replaced it with heating tape with a total capacity of 3700 W.

Run #7

Post

- All inside and outside thermocouples on the vessels and Centrax separator were interlocked through their controllers to prevent overheating of either inside of outside heating surfaces,

- to further improve temperature control of the bottom part of the heated section of the Centrax separator a heater with self-contained thermocouple was installed in the drainage channel of the flange connected to the top part of the drive mechanism housing,

- temperature control on the rest of the system was also improved by the addition of temperature control loops to specific sections of piping and to particular places of the process vessels,

- the Truarc bottom bearing retention ring (phase II reports, drawing no. 4053-102) was removed as its diameter increased under the centrifugal force which caused it to rub against the installed thrust load spacer ring. The function of this Truarc ring of containing the bottom bearing in its housing had become obsolete after installation of the thrust load assembly.

Run #8

Post

- Experienced continued problems with the turbine process flow meter on the slurry feed line due to failure of the electronics, it was corrected by the installation of a N_2 purge line to the inside of the signal transmitter housing of the flow meter,

- a relay was replaced in the position indicator controller to correct failure of this unit during run 8,

- to prepare for slurry feed rates in excess of 1 L/min in subsequent runs the FMI feed metering pump (maximum capacity of 1 L/min) was replaced with a FH32 Roto-King gear pump which has capacity well in excess of that called for in the experimental program.

Run #9

Post

- Due to wear and tear some of the heat tracing on the process piping had to be replaced,

- the Roto-King recycle pump of vessel number one was reconditioned, it showed significant wear with the carbon rotor shaft bushings worn more than 50%; both were replaced with bronze bushings; the stator was worn but not bad enough to warrant replacement.

Run #10

Post

- The micro-flow controller still failed at certain times which was found to be due to "noise" in the power supply to the unit; this problem was corrected by the addition of spike protection circuitry.

The improvement in operability of the Centrax pilot plant was very pronounced in going from run number one to run number ten. Run number ten was the last test run of this project.

Operational problems were, in essence, restricted to only three areas, namely:

- micro-flow controller and the process flow meters,
- temperature control of certain parts of the pilot plant, and
- proper operation of the bottom bearing of the Centrax separator.

Temperature control is due to the difficult task of operating at relative high temperatures. Attention to detail solves these problems as shown here. Instrumentation failures can be minimized by proper progression through and recognition of sections which require design improvements. Through the ten tests considerable progress was made in this area. The correction to the bottom bearing assembly is temporary. It needs to be redesigned for subsequent units.

Considering that this is the first prototype it ran very well. After implementation of the corrective measures as described the pilot plant was operated at design specifications.

This Centrax separator prototype is capable of operating at 180°C while its drive assembly attains a steady temperature of approximately 60°C which is well below the bearings operating temperature of 120°C. Additional test work is required to determine the maximum operating temperature. Because of the basic design feature of separate sections for processing and for the mechanical drive part of the unit, there is scope for very high temperature designs.

Operation at the relatively high speed of 4600 RPM presented no problems. Runs were terminated when the feed was exhausted, not because of operational difficulties.

Operating performance of this first prototype is thus exceptional in that it performed according to specifications. Discussed in the next chapter is the processing performance of this unit.

4.0 SEPARATION PERFORMANCE; PRELIMINARY RESULTS

The operational performance of the Centrax separator and the pilot plant as a whole is discussed in the previous chapter. Presented in this chapter are the results of the processing performance of the Centrax separator.

The feed material for these tests, was obtained from the Wilsonville pilot plant facility free of charge through the efforts and co-operation of EPRI personnel. It consisted of one barrel of atmospheric flashed slurry product from their coal liquefaction reactor, and one barrel of the bottoms of solids free hydrotreated liquid product. The former was used in this test work as the feed and the latter as extractant.

The composition of these two type of materials is as shown below:

TABLE 4.1 FEED AND EXTRACTANT COMPOSITION

COMPONENTS	FEED (wt %)	EXTRACTANT (wt %)
Oils	51.09	100
Asphaltenes	23.59	-
Pre-Asphaltenes	16.05	-
Solids	9.32	-
Unconverted Coal	4.45	-
Ash	4.87	-

The extractant is a liquid at room temperature with low viscosity. The feed, on the other hand, is a solid at room temperature. Its softening point is about 80°C. At approximately 120°C it is a liquid with its viscosity decreasing with increasing temperature. During the phase change from solid to liquid it is very difficult to process since it goes trough a "putty" type of transformation. This presents special problems in heating and transferring of this type of material as discussed in chapter 3.

4.1 Objective of Test Program

Since the Centrax separator was specially designed for processing and separation of solids from the above described "heavy" hydrocarbon slurries, it was tested with just such materials.

The specific objectives were to determine:

A. Quantitative:

- The separation capabilities of the apparatas under different conditions as measured by:
  - the clarity or the amount of ash removed from the separated liquid, and
  - the compaction of the separated solids.

B. Quantitative:

- The processing or transport capabilities of the separator with regard to:
  - the feed material entering into the unit,
  - the separated liquid leaving by gravity flow, and
  - the separated solids cake which is positively ejected from the separation zone into the receiving section of the separator.

4.2 Experimental Program

To determine the quantitative and qualitative parts of the objective a program of ten runs was laid out. For each run the dependent variables, percent ash in the separated liquid and the compactness of the separated solids, were measured. These provided the quantitative information as a function of the following selected independent process variables:

- feed flow rate,
- G force,
- composition or "type" of feed, and
- operating sequence of the scraper.

Runs were carried out with different values of each one of these variables or of a combination of them. Specific values were set based on prior experience of separating this type of material on a bench scale.

Inspection of different parts of the separator provided the information required for the qualitative aspects of the objective. At the end of each run the unit was partly dismantled to gain access to parts of the equipment which are normally hidden from direct view.

4.3 Typical Run Procedure

Although, the operation of the Centrax pilot plant is described in detail in chapter 2, the experimental details related to the collection of data are described here for clarity. The same procedure, in essence, was followed for each run. Minor modifications were introduced throughout the program to either obtain more accurate data and/or take additional measurements.

After the start of the feed a log-sheet was kept on which the values of the different independent variables were recorded at selected time intervals. A record of this is presented in Appendix 1. Also, throughout the run samples were taken of the separated liquid, and the feed. These samples were sent to the laboratory for immediate analysis. They were analyzed for precentage ash and in many cases, also for the percentage of solids, pre-asphaltenes, asphaltenes, and oils.

At the end of a run samples were collected from vessel number three which contained the separated liquid, and from vessel number four if the run had been carried out in the slurry mode. That is, if the separated cake was collected in extractant and re-slurried this was called the slurry mode as apposed to collecting these solids in an empty trough (Figure 2.4). This was denoted as operating in the solids mode.

The composition of the separated solids was obtained from samples taken either from vessel number four which contained the re-slurried solids or from samples taken from the Centrax trough in case the solids were not re-slurried. Additional solids samples were obtained by scraping sections of retained cake at the end of the run from the bowl wall and from the area under the bowl barrier (Figure 2.4). Analysis of these samples was identical to analysis of the feed and separated liquid samples.

For some of the runs, additional samples of the feed were obtained, which were then separated according to the previously developed bench-scale separation procedure (Phase I Report). This allowed for a direct comparison between the separating capabilities of the pilot plant Centrax unit and separation under ideal bench scale conditions. Results of these sub-tests and of the ten runs are presented and discussed next. The analytical procedure used for analysis of these samples was developed in phase I and is reported in the above mentioned document.

4.4 Test Results

The test results of the runs are presented in detail in Appendicies 1 and 2 Appendix 1. lists the operating conditions. Analyzed data are presented and discussed in the following sub-sections.

4.4.1 Ash Content of Separated Liquid

The effect of different operating variables on the clarity of the separated liquid was investigated.

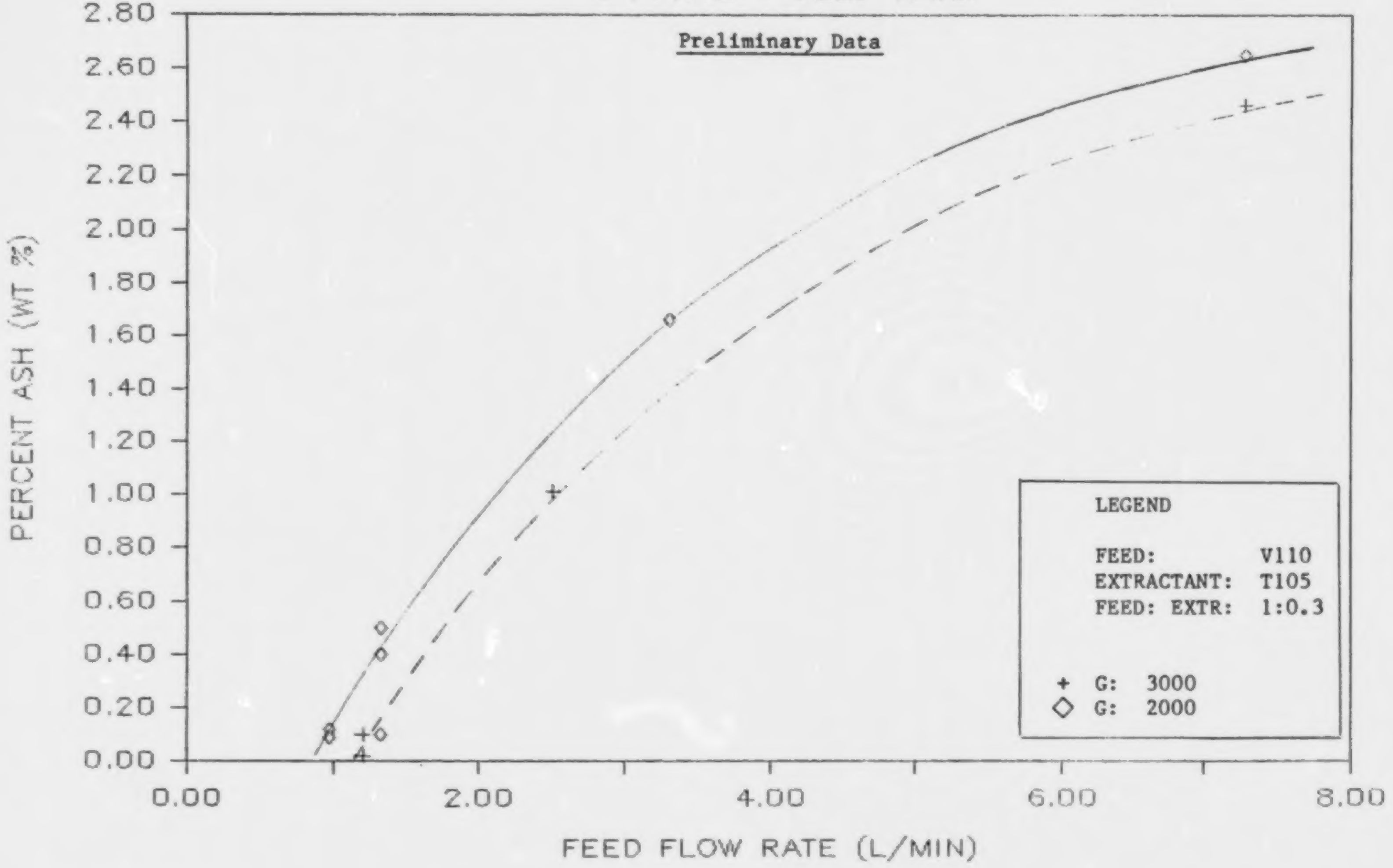
G Force

Shown in Figure 4.1 is the weight percent ash in the separated liquid as a function of feed flow rate at two levels of applied G force. The ash content of the feed was 3.78%, while the feed was made up of so called V110 material, which is the slurry product out of the coal liquefaction reactor, to which was added T105 liquid in a ratio of 1 to 0.3. That is, one part of V110 and 0.3 parts of T105. T105 liquid is the bottom of hydro-treated coal liquefaction product.

Application of a larger G force improves the clarity of the separated liquid. Although, as shown by the solid line, 2000 G's, and the dashed line, 3000 G's, the effect appears to be minor as compared to the effect of an increase in the flow rate of slurry being fed to the unit.

FIG 4.1 ASH CONTENT SEPARATED LIQUID

G FORCE - FEED RATE



At larger flow rates separation is poor for this type of feed material, and increasing the G force does not markedly improve this. However, at flow rates of the order of 1 L/min the application of a larger G force appears to be beneficial on the requirements of the ash content in the separated liquid. It may be a trade off between lower feed rates and higher G forces. The limited data presented here suggests that for specific applications this interaction needs to be investigated and optimized based on the cost of larger or more units versus the cost of fewer high speed separators.

Type of Feed

Figure 4.2 shows that the processing of different types of feed results in quite different performance characteristics as a function of feed flow rate for the same G force. The dashed line shows the effect of the type of feed described in the previous sub-section, that is a mixture of V110 and T105 with 1 part of V110 plus 0.3 parts of T105, while the solid line represents the results of separating the separated liquid of the above feed mixture. The average ash content of the separated liquid was 1.28%. It was collected in vessel number three and consisted of the separated liquid obtained at different feed rates and G forces as described. This liquid was pumped from vessel number three to vessel number one, from where it was used as feed for the subsequent run. The separation results of which are shown in Figure 4.2 as the squares.

These points show no apparent trend as a function of feed rate. They do, however, point toward a very definite correlation between separator performance and the characteristics of the feed material.

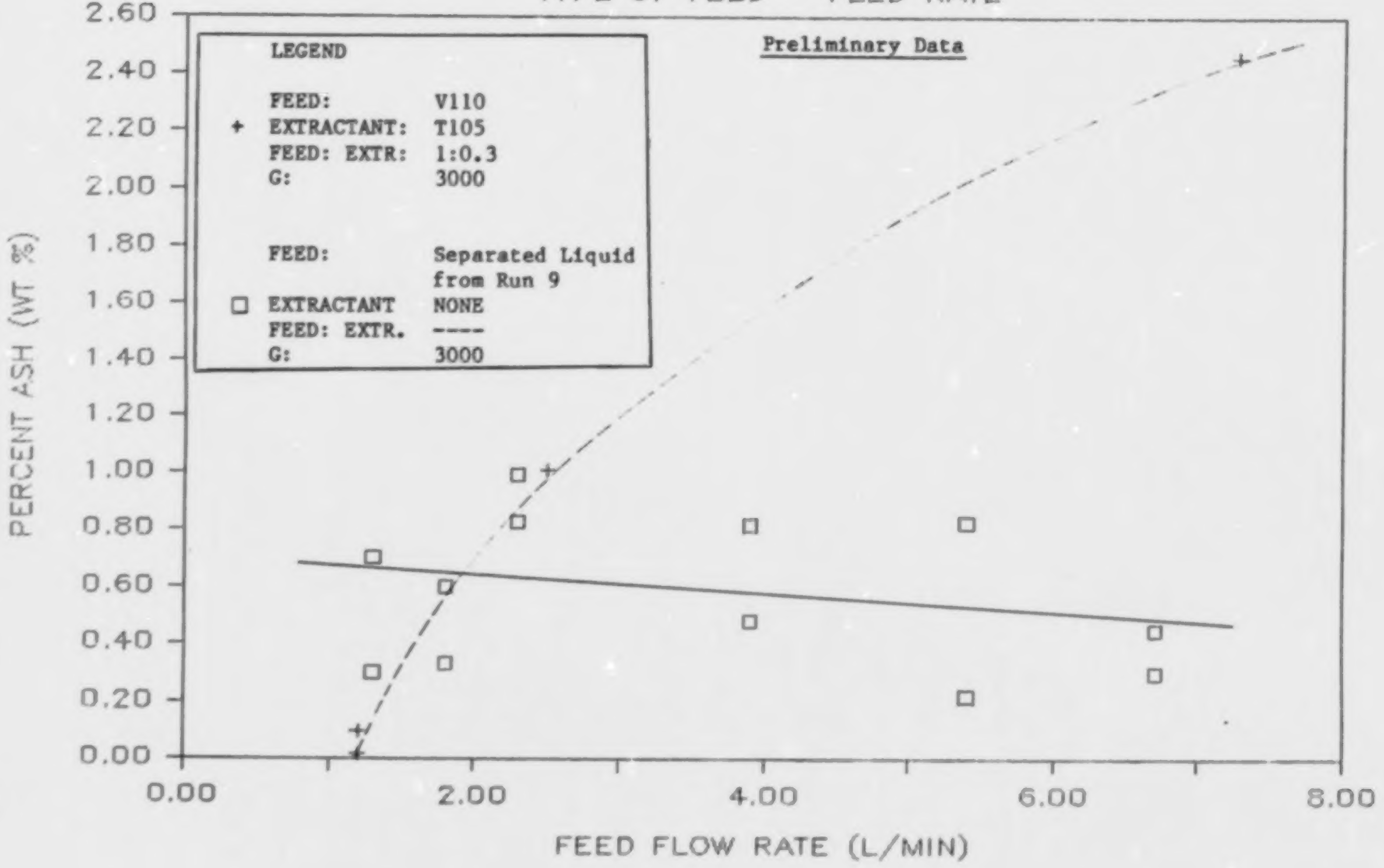
Since the composition of the liquid part of the feed slurry is the same for both feeds, it suggest that the radically different results are due to differences in the properties of the solids in these feeds. The properties of interest are probably, the average density, since the particles may vary in composition from unconverted coal to ash and any mixture of these, and the particle size distribution. Both parameters are known to have a significant effect on separation by centrifugal force. To clarity the results presented in Figure 4.2 any further these parameters need to be measured. This is beyond the scope of this project and therefore the effects shown in Figure 4.2 can only be inferred based on known separation characteristics of this type of separator.

Feed Rate

Both Figures 4.1 and 4.2 indicate that the feed rate has a definite effect on the performance of the separator. Again, this is in line with expectations, since separation is a function of the residence time of each particle in the separation zone of such an apparatus.

FIG 4.2 ASH CONTENT SEPARATED LIQUID

TYPE OF FEED - FEED RATE



The effect of this variable is confounded with the effects of G force and type of feed as shown on these figures. However, for the conditions were its effect appears to be dominant it shows that separation performance decreases with increasing feed rate. This is expected to be a direct result of the shorter time available for "capture" by a solid surface of each particle or cluster of particles. In addition, at the higher feed rates the local liquid velocities are higher which may result in re-slurrying of partial separated particles. This is a complex subject and is related to the fluid dynamics in the separation zone and the physical properties of the solid particles in combination with the effect of solids concentration.

Scraper Operation

Experiment number 10 was carried out to determine if the operation of the scraper had any effect on the ash content of the separated liquid. Since solids were found in the liquid discharge channel (Figure 2.4) of the Centrax separator after the last couple of runs, it was suggested that these solids may either end up in this place due to carry over with the liquid at high feed rates or be pushed through the liquid overflow parts by the solids scraper.

To resolve this, experiment number 10 was run in a particular sequence of intermittant feed coupled with the operation of the scraper. A sequence consisted of start of slurry feed, maintenance of feed while taking samples of the separated liquid, stop of feed for a certain time span during which the contents of the bowl remained in place, and lastly activation of the scraper which removed the liquid and solids out of the bowl and the taking of a sample during the upward motion of the scraper.

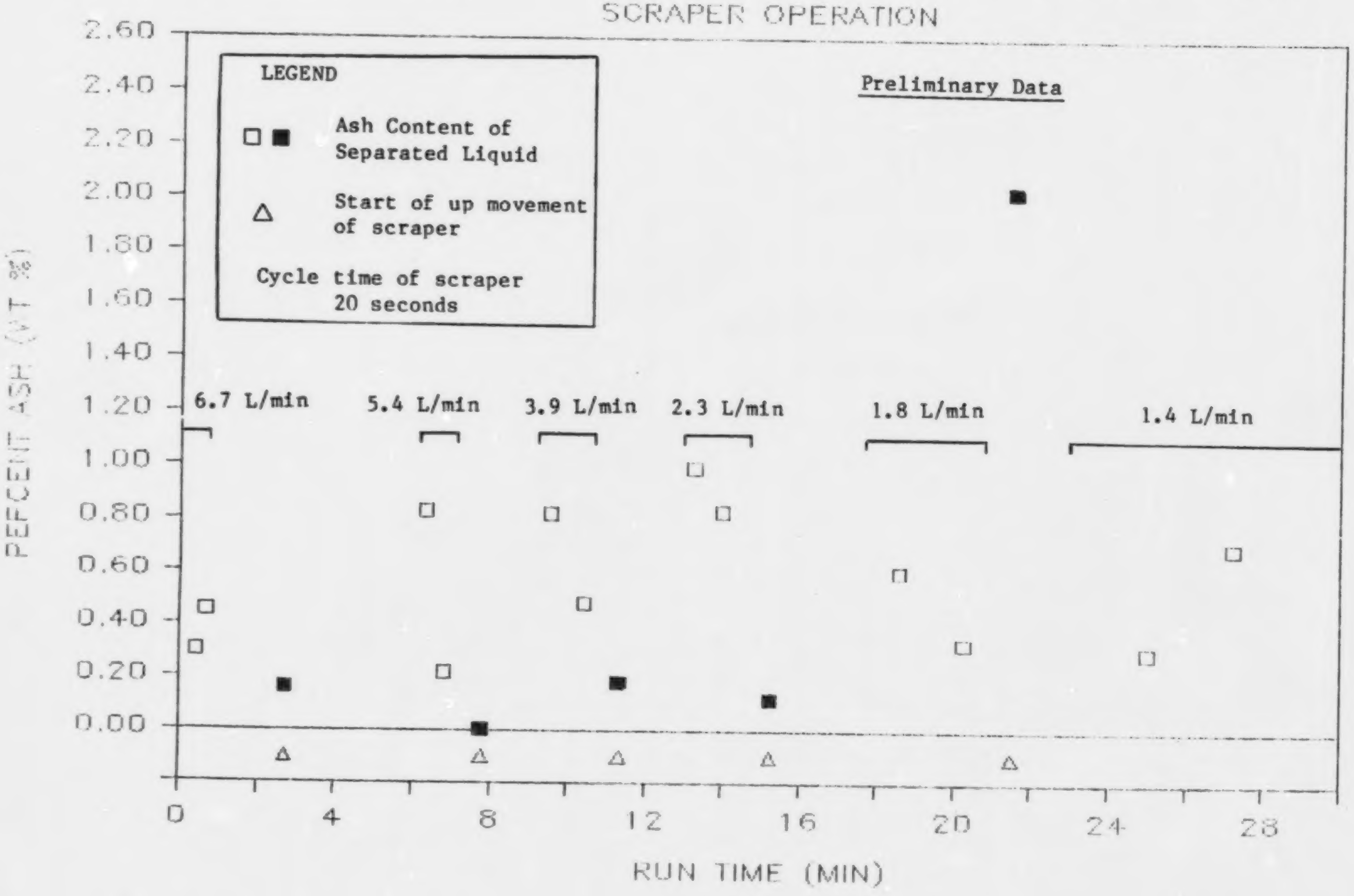
This sequence was repeated for different feed rates as shown in Figure 4.3. The speed of the separator was kept at 4600 RPM (3000 G's) throughout the operations of feeding and scraper up and down movements, as was done for all other runs.

The time span between stoppage of the feed and the start of the up movement of the scraper served to remove most of the solids from the liquid layer in the bowl. This is shown by the low ash content of the liquid pushed out of the bowl by the scraper denoted by the filled in squares of Figure 4.3. The ash content during feeding at different rates are shown by the open squares, which are discussed in the previous sub-section.

The low ash content of the first four filled in squares would suggest that during the pushing action of the scraper very little of any solids are pushed through the liquid overflow parts. However, the fifth point is the revealing piece of information with regard to this question of solids being pushed through these ports. The high value is not due to experimental error since during sampling it was observed that a "slug" of solids came through with the liquid. In addition, upon inspection at the end of the run solids were found in the liquid discharge channel.

FIG. 4.3 RUN 10 ASH CONTENT

SCRAPER OPERATION



Based on this preliminary data it is suggested that during each upstroke of the scraper a small amount of solids are pushed through the liquid overflow ports while the bulk of the solids are removed along the intended path over the bowl barrier. Another possibility is that the movement of the scraper causes the re-slurrying of some of the solids which then end up in the liquid discharge circuit. This is considered to be less likely than the possibility of the solids actually being pushed by the "squeezing" action of the scraper trough those ports. The four filled in low points and the sub-sequent very high point might indicate that the solids which are pushed through the ports are deposited in the liquid discharge channel, and after considerable build-up in this channel fractions of this deposit are occassionally carried along with liquid.

Given that the above arguments are correct it indicates that this section of the separator needs to be redesigned to minimize or eliminate this problem of "solids cross-over" into the liquid flow path. It is a particular problem related to the physical properties of the cake. This particular cake material has the consistency of a very viscous liquid or thin paste and serves to maximize this problem. An incompressible cake would probably show very little cross-over.

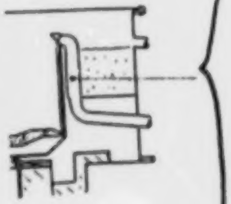
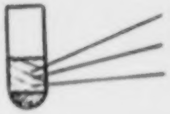
Comparison With Bench Scale Separations

During runs 7, 8, and 9 simultaneous bench-scale separations were carried out. Samples were taken from the feed to the Centrax which were immediately transferred without cooling to the pre-heated laboratory separator and separated. Details of the bench scale separations are presented in Appendix 3.

Shown in Table 4.2 are the results of the separation performance of the Centrax and the results of the laboratory separator for identical feeds. These are the results from Run #7 which are used here for discussion purposes. The comparative results from runs #8 and #9 are similar to those of Run #7 and are not presented in the body of the report since they do not add to the observations. As mentioned, all the results are, however, presented in Appendix 3.

It is seen that the composition of the separated liquid is within experimental error the same for both type of separations. That is, the concentration of oils, asphaltenes, and pre-asphaltenes are not different. Severe temperature differences during the two type of separations might have resulted in different concentrations due the effect of temperature on precipitation of asphaltenes and pre-asphaltenes. This was not observed in line with the temperature measurements during pilot and bench scale operations.

TABLE 4.2 RUN 7 SEPARATED LIQUID CONCENTRATIONS - PILOT PLANT VERSUS LABORATORY SEPARATIONS

CONDITIONS	LOCATION	COMPOSITION				
		Oil	Asphaltenes	Pre-Asphaltenes	UC	Ash
CENTRAX						
- Feed (average of 3 samples) - Contains ~ V110-247 - T105-247 (1:0.4)		62.39	20.31	11.93	2.81	2.57
- Sample #2 - before 1st stroke of scraper - Feed Rate 60 - 70 L/h - Tf = 180°C - Ts = 160°C		69.66	16.86	11.89	1.19	0.40
- Sample #3 - during 1st upstroke of scraper - Feed had been stopped for about 3 mins. - Ts = 165°C, Tf = 180°C - feed had been on for a total of 3 mins.		66.24	17.44	15.15	1.00	0.17
- Sample #4 - 2 min after 1st stroke of scraper - 1 min after restart of feed at ~ 56 L/h - Ts = 168°C, Tf = 180°C		70.99	16.35	11.33	1.17	0.16
- Sample #5 - 6 min after 1st stroke of scraper - 5 min after restart of feed at ~ 56 L/h - Ts - 170°C, Tf - 180°C		66.20	17.99	13.94	1.59	0.28
- Sample #6 - 2 min after 2nd stroke of scraper - 8 min aftr restart of feed ~56 L/h - Ts = 172°C, Tf = 180°C		65.24	20.54	12.95	1.13	0.14
Bench Scale						
#1		65.88	21.93	11.02	1.02	0.05
#2		68.31	19.22	11.62	0.79	0.06
#3		62.63	23.99	12.37	0.97	0.04

Note: Ts : Separation Temperature
Tf : Feed Temperature

There is, however, a marked difference in the ash concentration of liquid separated by Centrax as compared to that of liquid separated by the batch laboratory separator. The ash levels of the latter are consistently lower by a factor of 2 to 5. For example, ash concentration of separated liquid was:

- Centrax 0.17%,
- Bench Scale 0.05%.

This needs to be investigated in subsequent work. There are essentially two major differences between the two type of separators. One is the method whereby the particles are accelerated to the centrifugal separation speed, and the other is that the particles are subjected to different fluid dynamic forces while being separated. Improvements in the acceleration of the feed characteristics into the Centrax can probably be made by re-design and should be addressed in further work. After that fluid dynamics could be investigated if necessary.

4.4.2 Compaction of Separated Solids

Shown in Table 4.3 are typical results of the concentrations of the solids cake. Compaction is the term used to denote the amount of compression of the solid particles. The trapped liquid in between the particles is referred to as "interstitial" liquid. A cake consist thus of interstial liquid and solids compacted to, for instance, 40.58%. The 40.58 is the weight percent of solids in the cake. Solids in this work consists of unconverted coal and ash. The weight percent of oils (O), asphalaltenes (A), pre-asphaltenes (PA), and solids (S) are normalized to 100%. The percent ash is also normalized to 100% and is not the percentage of the solids. Thus for a composition as show in Table 4.3 of:

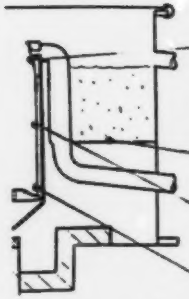
O	A	PA	S	Ash
32.24	6.85	20.33	40.58	27.68

it means that out of 100 parts, 40.58 are solids, 27.68 are ash and 40.58 - 27.68 - 12.90 are unconverted coal.

Rows 1 through 4 show the compaction results of the Centrax separator, while shown in rows 5 trough 7 are those obtained with the laboratory separator. Based on the ash measurements it is seen that the Centrax compacts the solids to the same or higher density than the lab separator.

These results are as expected. Solids are subjected to similar or higher G forces in the Centrax, and when they are pushed over the bowl barrier the scraper applies an additional force on the cake material. The feature of applying a compressive force on the cake while pushing it out of the separated liquid allows for the minimization of interstitial liquid. This has a very positive impact on the economics of separation.

TABLE 4.3 RUN 7 SEPARATED SOLIDS COMPACTION PILOT PLANT VERSUS LABORATORY SEPARATIONS

CONDITIONS	LOCATION	ROW #	COMPOSITION				
			Oil	Asphaltenes	Pre-Asphaltenes	UC	Ash
Pilot Plant							
end of run		1	32.24	6.85	20.33	12.90	27.68
		2	29.67	5.65	12.32	20.15	32.21
Tf = 180° Ts = 160 - 180°C		3	40.39	10.46	14.80	15.65	18.70
		4	34.52	17.42	3.53	21.06	23.47
		5	33.59	6.86	15.00	19.94	24.61
Bench Scale							
#1 Feed to Centrax Tf = 180°C Ts = 180°C		6	40.54	11.09	11.42	36.95 17.14	19.81
#2 Feed to Centrax Tf = 180°C Ts = 150°C		7	44.23	11.36	15.34	29.07 13.64	15.43
#3 Feed to Centrax Cooled to room temperature and pre-heated and separated next day. Tf=180°C Ts=180°C		8	40.06	15.23	9.21	35.50 16.14	19.36

Note: Tf : Feed Temperature
Ts : Separation Temperature

4.4.3 Qualitative Observations

At the end of each run the Centrax was partly disassembled for inspection. Throughout the program the following qualitative observations were made with regard to how well the Centrax transported the separated materials.

Separated Liquid

The separated liquid exited by gravity flow from the unit without any problems. The amount which was not pushed out of the bowl at the end of the run drained through the respective drain holes into receiver at the bottom of the heated section of the Centrax in after the bowl came to a full stop. Initial problems were experienced with this due to insufficient heating of some of the sections through which the liquid drained. Additional heating and temperature control solved this problem.

Depending on the control of the direction of flow of fumes and nitrogen gas, it was found that some of the separated liquid was carried as a mist into different parts of the heated section of the Centrax separator. Later tests showed that proper control over the dynamics of the gas phase eliminated this problem. Future designs should be modified to make the unit less sensitive to the dynamics of the vapour phase and provide better control over the direction of flow of the separated liquid when it exits from the rotating bowl into the stationary liquid discharge channel.

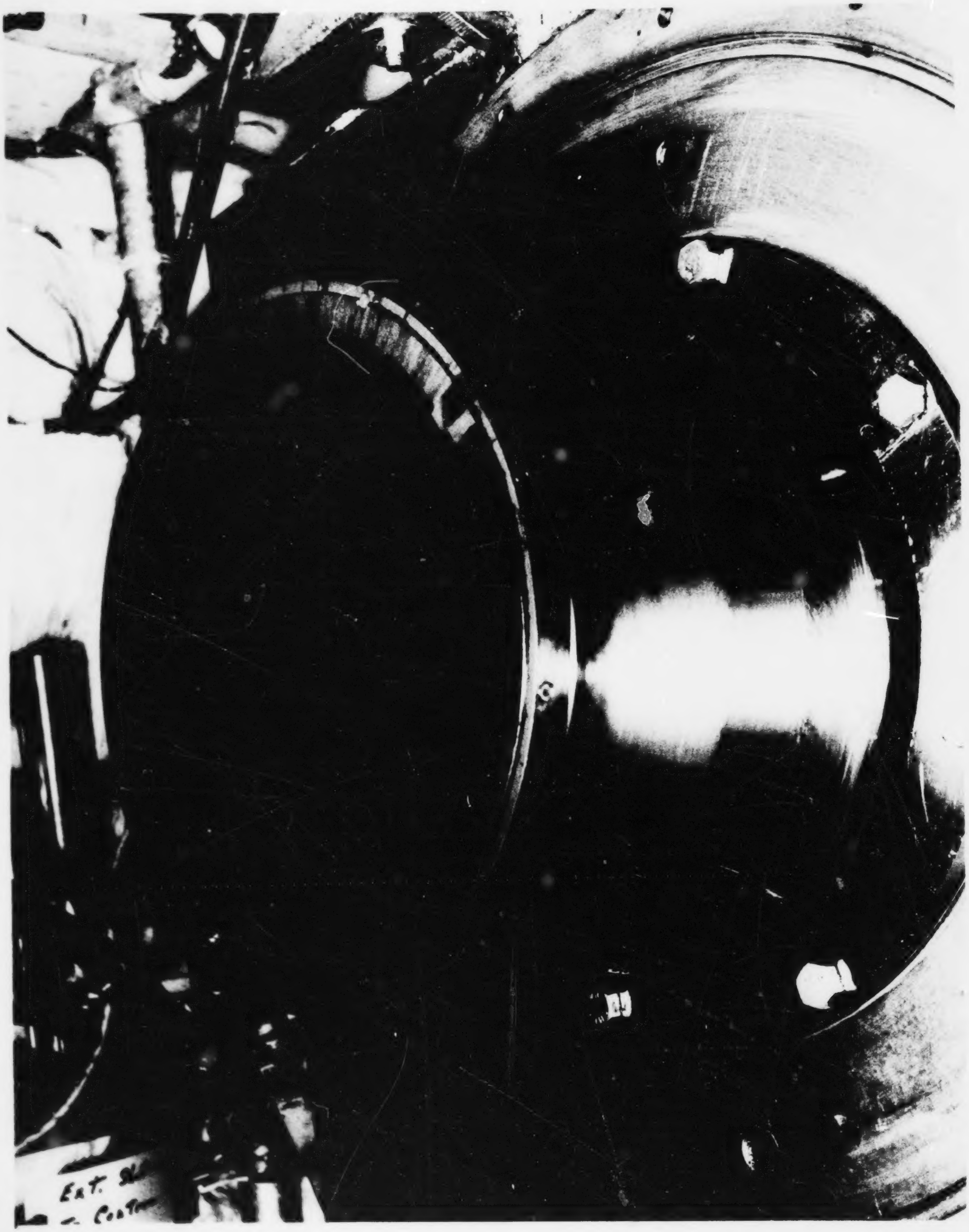
Separated Solids

The physical characteristics of this type of cake material dictated special requirements for transport. As mentioned in previous sections, this cake material has the consistency of a "very viscous fluid" or a putty at operating temperature, and secondly it has a tendency to "stick" to most surfaces. Because of its ash content the material is also very abrasive and can lead to excessive wear. Special design features were build into the Centrax to account for these characteristics.

They are described in detail in Chapter 2. It was found that solids did not accumulate at the area under the scraper. The sloped design of the bottom of the bowl provided for a constant upward and outward and any force solids which ended up in this section. Minimal build-up occurred to a steady-state level.

Shown in Figures 4.4 through 4.6 are typical cake formations. From Figure 4.4 it is seen that the cake thickness decreases in going from the bottom of the bowl towards the top. Larger and heavier particles are probably deposited in the bottom section since the feed is introduced at that point, while smaller particles end-up at the top.

FIG. 4.4 TYPICAL CROSS SECTION OF FORMED CAKE



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to Cont

FIG. 4.5 VIEW OF ESTABLISHED CAKE ON INSIDE OF BOWL WALL

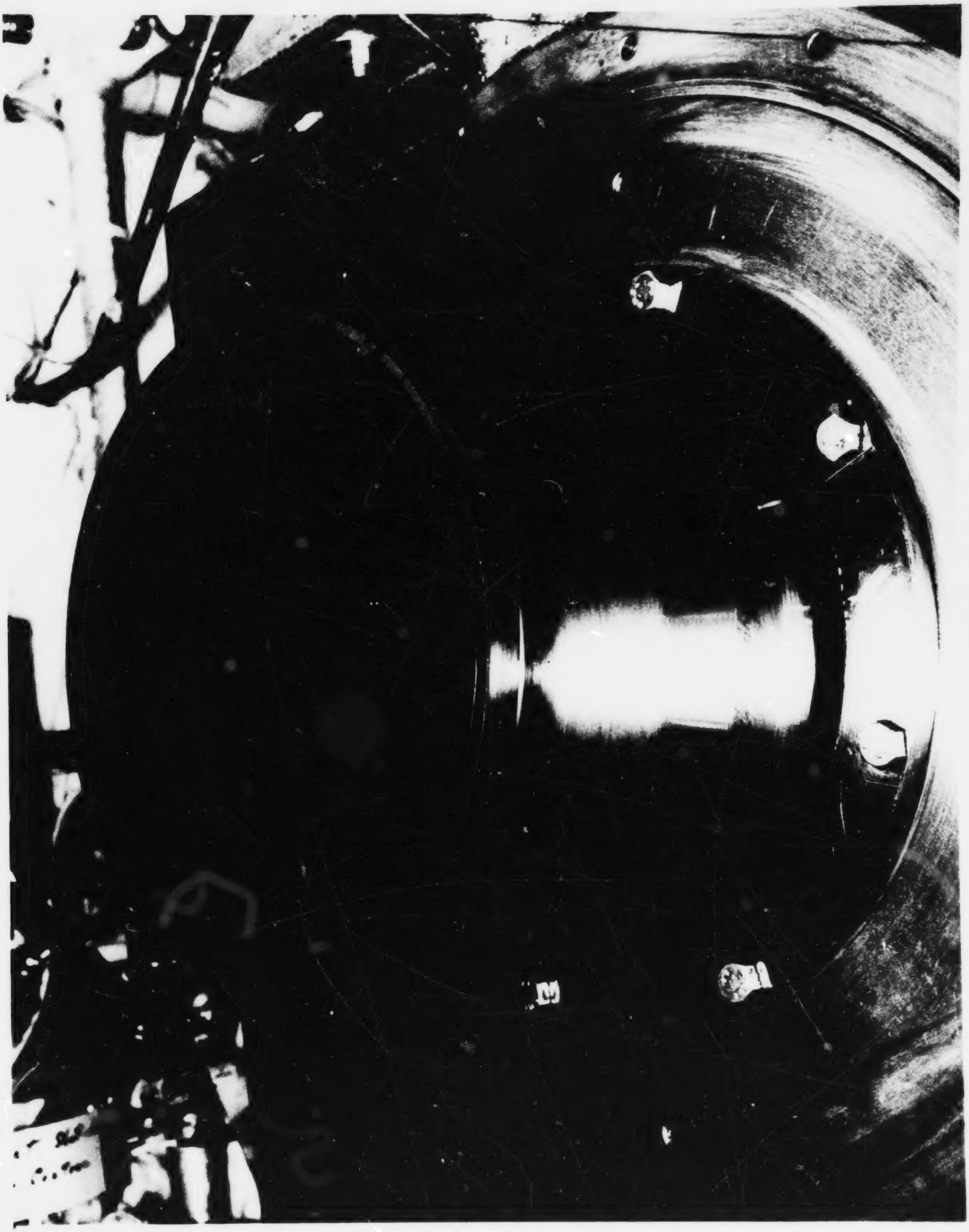
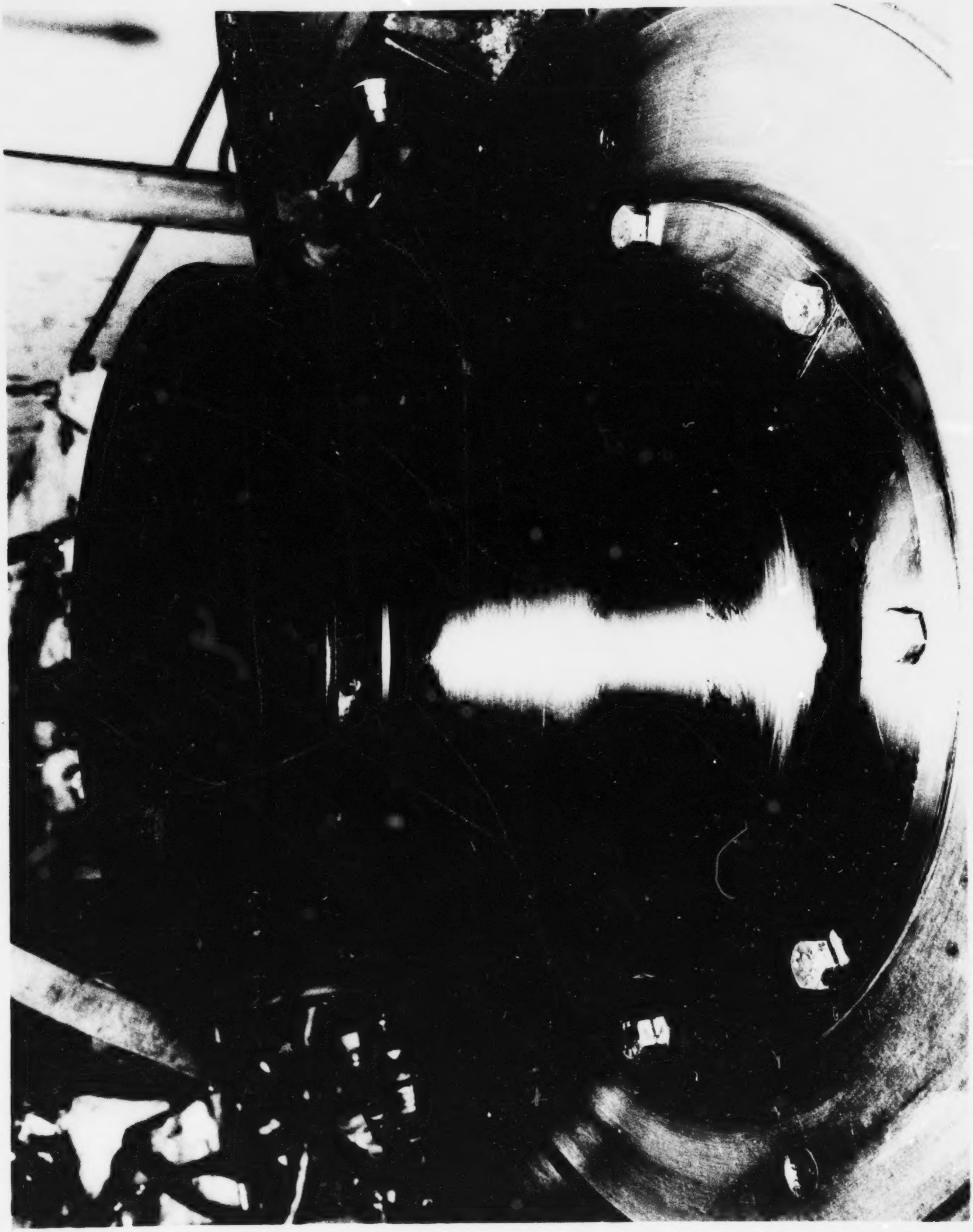


FIG. 4.6 CLOSE-UP VIEW OF LIQUID OVERFLOW PORTS



Figures 4.5 and 4.6 show very clearly that the liquid overflow ports remain open during processing. Any solids which are pushed into these holes during their discharge are probably ejected into the liquid discharge section. Although this leads to the problem of contamination of the separated liquid, as discussed in previous sections, it does not compound the processing difficulties by plugging of the liquid discharge holes.

Solids were ejected out of the bowl over the bowl barrier as designed. No build-up occurred on the solids deflector ring, and in the slurry mode the solids were re-slurried without any problems.

Abrasion was localized to the scraper wear ring since it was observed that a permanent one millimeter thick cake was formed on the bowl wall. The bowl wall was thus protected from abrasion by this deposited material. Again, this is according to design and one of the features of the Centrax separator.

4.5 Processing Performance

Quantitative measurements showed that the ash concentration of the separated liquid can be reduced to very low levels depending on the feed rate, applied G force, and the composition of the feed. However, separation of identical feed material on the pilot plant scale (Centrax) and on the bench scale showed that ash levels in the separated liquid were consistently higher by a factor of 2 to 5 for the former. For example, ash concentration of separation liquids was:

- Centrax	0.17%
- Bench scale	0.05%

This needs to be investigated further.

Preliminary data suggests that some of the separated solids are pushed into the exit lines of the separated liquid during removal of these solids from the separator. This "cross-over" of separated solids back into the separated liquid needs to be minimized or eliminate in subsequent designs. This problem is a strong function of the compressibility or plasticity characteristics of the solids cake, and this problem is at its maximum for this application. The cake has the consistency of a very viscous liquid at the processing temperature.

Qualitative observations showed that there was no build-up of liquids and solids in any part of the Centrax of their respective areas. Liquid exited by gravity and solids were pushed out of the bowl as designed. Build-up of solids under the scraper did not occur in line with the design concepts.

5.0 UPGRADING OF SEPARATED PRODUCT

The objective of this testing program was to investigate if the Centrax separation process has adverse effects on the upgrading of the coal liquefaction recycle liquid product.

Two sets of catalytic hydrotreating tests were conducted under the same experimental conditions using as feedstock the material separated from the solid/liquid coal liquefaction product, obtained from Wilsonville. This product was designated V110 bottoms, Run 247. In the first set of three tests the liquid feedstock used was obtained by separating the V110 bottoms by filtration using a milipore pressure holder. This material was designated as V110 Filtrate. In the second set of three tests the liquid feedstock used was obtained by separating the V110 bottoms using the CENTRAX separation process developed by SANDTECH. This material was designated V110-Centrax. The catalyst used in both sets of tests, was Shell 324M obtained from Wilsonville.

The extent of hydrotreatment was evaluated as follows.

- conversion of the 550°C + fraction into distillable products
- consumption of hydrogen
- yield of water
- yield of $C_1 - C_3$
- change in the oils, asphaltenes, pre-asphaltenes content.

The effects of the CENTRAX separation process on the upgrading of the coal liquefaction separated liquid product were evaluated by comparing the results, based on the above criteria, obtained in the hydrotreating tests using as feedstock the CENTRAX separated material with those using as feedstock the filtered material.

5.1 Description of Equipment

The catalytic hydrotreating tests were carried out in a hot charge/discharge batch mode. A schematic diagram of the apparatus used is shown in Figure 5.1.

The apparatus consisted of a liquid feed injection system, the reactor, a hot discharge receiver and a vapour phase recovery unit.

The reactor was a 2 litre, 316 stainless steel, stirred, electrically heated autoclave. The maximum operating pressure and temperature of the reactor were 34.5 MPa and 450°C respectively.

For the present experimental program this reactor was equipped with the following features:

- An Autoclave Engineers Magnedrive II, variable speed, magnetically actuated, packless impeller system.
- A stationary annular catalyst basket made of stainless steel mesh.
- Three internal baffles for increased mixing.
- Connections for liquid feed injection, gas inlet/outlet, thermocouple insertion and bottom discharge.
- Two thermocouples, connected to on/off controllers, dial temperature indicators and a multi-point recorder. One of the thermocouples was located in the catalyst bed and was used for controlling the heater and the other thermocouple was located on the outside reactor wall, and activated the heater shut-off at a preset temperature.
- Two electrical band heaters, 1500 watts each fitted around the reactor.
- A Bourdon-type dial pressure gauge, 0-35 MPa dial range with 0.5 MPa dial sub-divisions.

The hot discharge receiver was a custom made 300 ml, 316 stainless steel, high pressure vessel. This vessel was provided with electrical band heaters and temperature measurement and control.

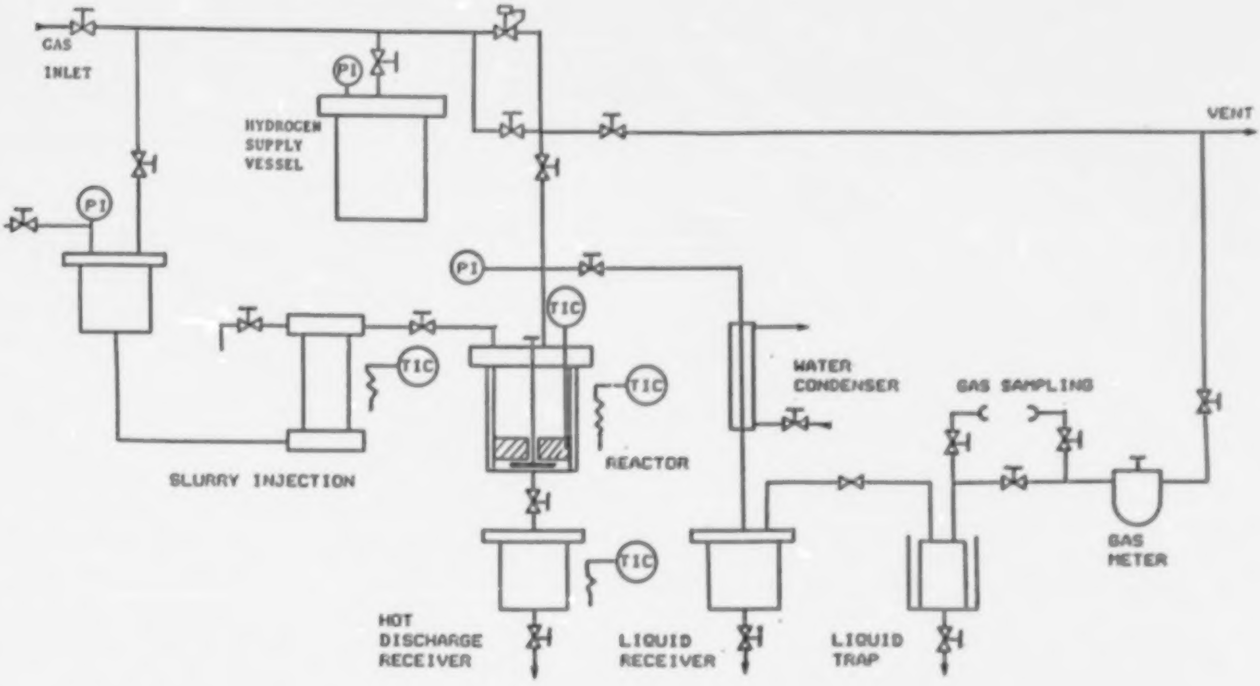
The liquid feed injection system was "in house" designed and included a hydraulic cylinder of 900 ml capacity, 34.5 MPa maximum operating pressure and 120°C maximum operating temperature. This cylinder was connected to a high pressure, 316 stainless steel vessel. This vessel, designated the "hydraulic vessel", contained hydraulic oil and was connected to the inert gas manifold. The liquid feed slurry drawn into the cylinder was injected into the reactor by applying to the "hydraulic vessel", an inert gas pressure higher than the pressure in the reactor.

The vapour phase recovery unit consisted of a tubular water condenser, a light liquid receiver, a refrigerated trap, a gas sampling arrangement and a wet-test gas meter. The off gas was discharged via a scrubbing tower. The light condensed liquid receiver was a 1-litre, 316 stainless steel, high pressure vessel. The refrigerated liquid trap was a 300 ml capacity, 316 stainless steel vessel with a cooling mantle.

The gas sampling arrangement allowed for thorough purging of the gas sampler tube before a sample was taken.

The wet-test gas meter was a "Precision Scientific", model 63126, 3.0 litres dial range with 0.01 litres dial sub-divisions.

FIG. 5.1 SCHEMATIC DIAGRAM OF HOT CHARGE/DISCHARGE REACTOR SYSTEM


GAS INLET
PI
HYDROGEN SUPPLY VESSEL
VENT
PI
PI
TIC
TIC
TIC
SLURRY INJECTION
REACTOR
WATER CONDENSER
GAS SAMPLING
GAS METER
TIC
HOT DISCHARGE RECEIVER
LIQUID RECEIVER
LIQUID TRAP

5.2 Testing Procedures

5.2.1 Feedstock Preparation

The V110-CENTRAX feedstock was obtained from Run 6, and taken from vessel 3 and is a representative sample of the separated liquid obtained during the first 21 minutes of run time.

The V110-Filtrate feedstock was obtained by performing three batch filtrations using a 500 ml millipore pressure filter holder. The filtrations were carried out at 200°C and 150 psi pressure using a Whatman fiber glass filter.

5.2.2 Catalyst Preparation

The catalyst used in both series of tests was Shell 324M obtained from Wilsonville. The catalyst was presulphided at the Wilsonville facilities. It was received in the shape of extrudate submerged in process oil. In each series of tests fresh catalyst was used. In preparation for the tests the process oil was drained and the catalyst was then thoroughly washed with THF. The THF was subsequently removed from the catalyst by evaporation under vacuum. The dried catalyst was packed in the annular backet, before each set of tests, and placed in the reactor.

5.2.3 Testing

Before each test, the unit was pressure tested to 1.5 times the reaction pressure with inert gas H_2/N_2.

The reactor was then purged with hydrogen and pressurised with hydrogen to a predetermined cold pressure. The reactor was gradually heated to about 20°C below the desired reaction temperature. Both the inside and outside temperatures of the reactor were continuously recorded. The pressure of the reactor was recorded at regular intervals.

The liquid feed was heated to 120°C in order to ensure sufficient fluidity for injection. The hydraulic cylinder was maintained at 100°C.

The liquid feed was then drawn into the hydraulic cylinder and immediately injected into the reactor. The controllers of both the inside and outside reactor temperatures were manually adjusted immediately after injection in order to avoid an over-shoot in the temperature of the catalyst bed. The desired reaction temperature was reached within 3-6 minutes after injection. The temperature of the catalyst bed was then maintained constant during the length of the reaction time. The pressure in the reactor was also maintained constant during the length of the reaction time using a forward pressure regulator installed in the line connecting the reactor to a hydrogen supply vessel.

Upon completion of the reaction time, the liquid product was discharged into the hot discharge receiver which was kept at 150°C to maintain the fluidity of the liquid product.

The vapour phase was vented through the vapour phase recovery unit. The non-condensible gases were metered on the wet-test meter and analysed by gas chromatography before being discarded. The liquid products which condensed from the vapour phase were collected in the liquid receiver and liquid trap.

The entire unit was finally purged with inert gas. The products collected in the hot discharge receiver, liquid receiver and liquid trap were then recovered and weighed. The reactor and the hot discharge receiver were allowed to cool to room temperature. Both vessels were then washed with THF to recover the heavy product which remained in the vessels and the catalyst bed. The liquid product carried in THF was recovered from THF in a rotary vacuum evaporator. The weight of this product, designated as "washings", was used in the calculation of the material balance.

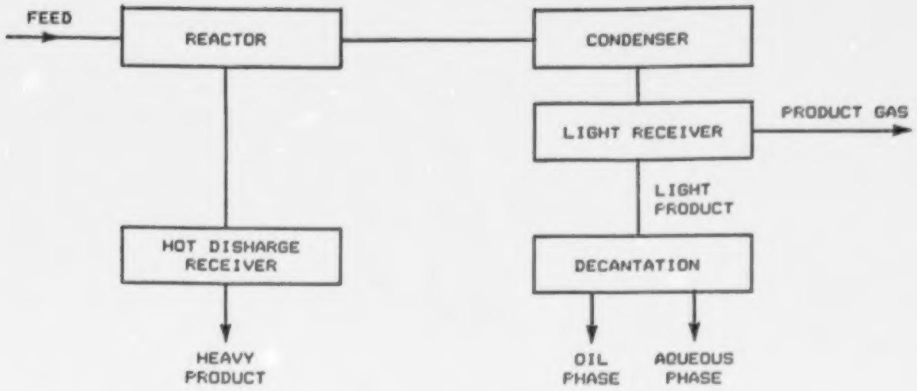
The THF was removed from the catalyst in the basket by vacuum.

5.2.4 Product Recovery

The products were recovered in three streams, as shown in Fig. 5.2. The product collected in the hot discharge receiver was designated "heavy product". This product was solid at room temperature. The vapour phase products were recovered in two streams: the light product and the gas product. The light product stream recovered from the liquid receiver and liquid trap contained water and organic liquids formed during the reaction time. This stream consisted of two different layers. The layers were separated by decantation. The top oil-like layer was designated the oil phase and the bottom water-like layer was designated the water layer.

The product gas stream was recovered by measuring the volume of the non-condensable gases on the wet-test gas meter and analysing the gas sample by a Carle gas chromatograph. This stream composed of H_2, CO_2, CO, H_2S, CH_4, C_2H_6, C_3H_8.

FIG. 5.2 PRODUCT STREAMS


FEED
REACTOR
CONDENSER
LIGHT RECEIVER
PRODUCT GAS
LIGHT FRODUCT
HOT DISHARGE RECEIVER
DECANTATION
HEAVY PRODUCT
OIL PHASE
AQUEOUS PHASE

5.3. Analytical Procedure

5.3.1 Material Balance Calculation

The material balance was calculated on percent of total input.

The weight of liquid feed introduced into the reactor was obtained by difference of the weight of the container before the injection less the weight of the container after the injection and drainage of the lines back into the initial container. The output was calculated by summing the weights of the heavy product collected in the hot discharge receiver, the light product collected in the liquid receiver and ice trap, the washings after the solvent was removed by evaporation and the non-condensable gases. The latter was calculated from the STP volume of the gases and the chromatographic analysis.

5.3.2 Analysis of Feedstocks

The analytical procedures used to analyse both the feedstocks and products are outlined in detail in Appendix 5.

The liquid feedstocks used in both sets of tests were analysed for selective solvent extraction with hexane, toluene and tetrahydrofuran (THF) to determine the maltenes, asphaltenes, pre-asphaltenes and solids, defined as follows.

Solids : THF Insolubles
Pre-asphaltenes : THF Solubles - Toluene Solubles
Asphaltenes : Toluene Solubles - Hexane Solubles
Maltenes : Hexane Solubles

A homogeneous sample of the liquid feedstocks was subjected to vacuum distillation under 0.1 mmHg vacuum to 550°C equivalent atmospheric pressure boiling point.

5.3.3 Analysis of Products

The heavy product was analysed for selective solvent extraction with hexane, toluene and THF to determine the maltenes, asphaltenes pre-asphaltenes and solids.

A representive, homogeneous sample, consisting of prorated amounts and heavy product and light oil product was subjected to vacuum distillation under 0.1 mmHg vacuum to 550°C equivalent atmospheric pressure boiling point.

The product gas stream was analysed on a "Carle" gas chromatograph to determine the percentage volume of H_2, CO_2, CO, C_1, C_2, C_3, H_2S.

5.4 Results and Discussion

Three tests were conducted in each set under the same experimental conditions as shown in Table 5.1. Table 5.2 presents the material balance calculations for all tests.

TABLE 5.1 EXPERIMENTAL CONDITIONS

Temperature	:	360°C
Pressure	:	18.6 MPa (2700 psig)
Time	:	60 Minutes
Mixing	:	800 rpm

TABLE 5.2 MATERIAL BALANCE CALCULATIONS

Feedstock (g)	Test 1.	Test 2.	Test 3.	Test 4.	Test 8.	Test 9.
Liquid Feed	382.7	366.5	369.5	316.4	329.4	323.9
Hydrogen	21.2	17.7	17.5	14.4	14.2	15.7
Total Feed	403.9	384.2	387.0	330.8	343.6	339.6
Product (g)						
Heavy	297.5	281.6	341.6	238.4	303.1	272.0
Light Organic	42.8	43.7	27.6	30.7	9.0	22.3
Light Water	N.D.	17.3	6.8	12.4	7.8	9.3
Washings	32.7	5.3	0.0	21.3	13.0	26.9
H_2	11.2	8.4	9.3	9.8	11.6	10.6
CH_4	0.9	0.4	0.2	1.3	0.0	0.0
C_2H_6	0.3	0.2	0.2	0.3	0.2	0.2
C_3H_8	5.3	19.0	6.3	2.5	1.7	1.2
H_2S	0.5	0.0	0.0	1.2	0.2	0.3
Total Product	391.2	375.9	392.0	317.9	346.6	342.0
Material Balance (% wt)	96.9	97.8	101.3	96.1	100.9	100.7

The selective solvent extraction analysis of the coal liquefaction solid/liquid product V110 Bottoms, Run 247 received from Wilsonville are presented in Table 5.3.

TABLE 5.3 PROPERTIES OF V110 - BOTTOMS, RUN 247

Selective Solvent Extraction (% wt).

Maltenes	50.1
Asphaltenes	25.2
Pre-Asphaltenes	15.4
THF Insolubles	9.3

Table 5.4 presents the properties of the liquid feed V110-Filtrate which was separated from the V110 bottoms by filtration and used in tests 1, 2, 3 and the liquid feed V110-CENTRAX which was separated from the V110 bottoms by the CENTRAX separation process and used in tests 7, 8, 9.

The properties of the products obtained in all tests are shown in Table 5.5.

The conversion of the $550^{o}C$ + fraction of the liquid feed was calculated as follows:

$$\text{Conversion (\% wt)} - (1 - 555^{o}C + \text{fraction in Product}/550^{o}C + \text{fraction in Feed}) \times 100$$

The consumption of hydrogens, water yield, C_1C_3 hydrocarbons yield and hydrogen sulphide formation were calculated as percentage of the $550^{o}C$ + fraction in the feedstock.

The conversion of the $550^{o}C$ + fraction, the hydrogen consumption and yields are shown in Table 5.6.

TABLE 5.4 PROPERTIES OF LIQUID FEEDS

	Selective Solvent Extraction (% wt).					
	V110 - Filtrate			V110 - Centrax		
TEST #	1	2	3	7	8	9
Maltenes	57.5	55.3	54.5	58.2	58.2	58.2
Asphaltenes	29.3	28.4	26.9	22.2	22.2	22.2
Pre-Asphaltenes	12.1	14.9	17.0	16.0	16.0	16.0
Solids	1.1	1.4	1.6	3.6	3.6	3.6
Vacuum Distillation (% wt)						
550°C - Fraction	45.9	45.9	45.9	48.6	48.6	48.6
550°C + Fraction	54.1	54.1	54.1	51.4	51.4	51.4

TABLE 5.5 PROPERTIES OF PRODUCTS

	Selective Solvent Extraction of Heavy Product (% wt).					
	Heavy & Light Organics			Heavy		
TEST #	1	2	3	7	8	9
Maltenes	82.4	67.8	68.2	82.5	78.5	76.2
Asphaltenes	15.2	28.4	25.9	14.1	27.1	19.1
Pre-asphaltenes	1.7	3.6	5.7	0.8	2.0	1.9
Solids	0.7	0.2	0.2	2.6	2.4	2.8
Vacuum Distillation (% wt)						
550°C - Fraction	59.7	58.5	51.5	59.2	57.8	53.5
550°C + Fraction	40.3	41.5	48.5	40.8	42.2	46.5

TABLE 5.6 CONVERSION, HYDROGEN CONSUMPTION AND YIELDS

		V110-Filtrate			V110-Centrax		
Test #		1	2	3	7	8	9
Conversion $550^\circ C$ +		27.4	30.8	10.4	34.8	21.2	16.5
H_2 Consumption		4.8	4.7	4.1	2.8	1.5	3.1
Yield	Water	N.D.	8.7	3.4	7.6	4.6	5.6
	C_1-C_3 Hydrocarbons	3.1	9.9	3.3	2.5	1.1	0.8
	H_2S	0.2	0.0	0.0	0.7	0.1	0.2

A comparison of the results in tables 5.5 and 5.6 indicate that similar patterns were obtained in the two sets of tests for the $550^\circ C$ + conversion, hydrogen consumption, yields, and selective solvent extraction. Although all tests were carried out at the same conditions the conversion of the $550^\circ C$ + fraction decreased with catalyst age in each set of tests.

Due to the limited number of tests, the causes of catalyst deactivation were not determined in this testing program. The results, however, determined with fresh catalyst in tests No 1 and 7 appear to be in agreement with the results obtained in the upgrading of same catalyst activity feedstocks (Technical Progress Report, Run 243, Illinois 6 Coal, Catalytic Inc., Wilsonville, Alabama, July 1983).

The comparison of the results in tests 1 and 7,Table 5.6, show that the upgrading of the CENTRAX separated material gave higher $550^\circ C$ + conversion, less hydrogen consumption, less C_1-C_3 hydrocarbon formation and better hydrodesulphurisation.

In test 7, the conversion of the $550^\circ C$+ fraction in the V110-CENTRAX feedstock was 34.8 wt % as compared to 27.4 wt% conversion of the $550^\circ C$+ fraction of the V110-filtrate in test 1.

The consumption of hydrogen was significantly less in test 7 (2.8 wt %) than that of test 1 (4.8 wt %).

The removal of sulphur from the feedstock as hydrogen sulphide was higher in test 7 (0.7 % wt) than that of test 1 (0.2 wt %).

Based on these results, it may be concluded that the separation of the liquid coal liquefaction product by the CENTRAX separation process does not present any adverse effects on the upgrading of the separated material.

6.0 ECONOMIC IMPLICATIONS OF CENTRAX DEASHING

The benefits of Centrax Deashing for the integrated two-stage liquefaction process are related to the overall economics of converting coal to usable liquid products. Mitre (1983) in a comparison of solvent deashing processes stated that in the

> "general range of deasher performance, each percent reduction in the rejection of liquid 850°F + material results in about a 1 percent reduction in the final product cost"

The Centrax Deashing Process has the ability to prevent any rejection of 850°F material, thus the process should offer further reductions in the final product cost. However, such a statement is simplistic in view of the many intereactions in coal liquefaction as a result of changes, or substitution, of any unit operation with another. The following section uses a complex economic model to predict the sensitivity of the integrated two-stage process to Centrax Deashing.

6.1 Economic Modelling-Coal Liquefaction

6.1.1 Economic Modelling Methodology

In the absence of a specific proposed project, with site, coal feed, desired product slate, pricing requires, etc, established, comparative economics must be made using models which generalize to a large degree. However, the model must be sufficiently complex to include all factors that can influence the financial return to the owners of coal liquefaction plants.

The following criteria are relevant for this analysis:

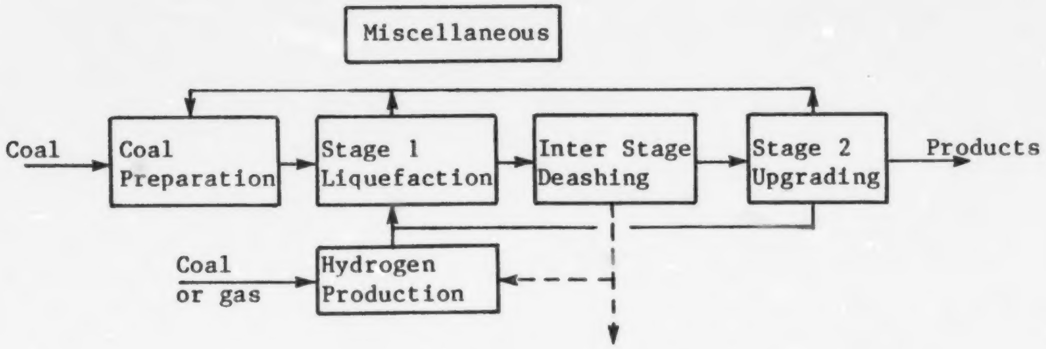
- The model must include the cost of producing coal liquids from incoming raw materials to final plant products.
- The economics over the life of the development must be considered, not just for the first year.
- Tax regimes and the owner's tax situation are too variable to be included.
- Capital costs must be developed for individual unit operations that will be affected by the deashing process employed.

This criteria has dictated the following approach and methodology for conducting a comparative economic benefit evaluation of deashing technologies:

- Development of a generalized coal liquefaction facility divided into major unit operations;
- Identification of process interactions between these major unit operations;
- Estimation of capital costs for individual major unit operations on a unit throughput basis;
- Incorporation of a range of technical, feed and product price assumptions, financing assumptions and inflation assumptions;
- Calculation of the economic viability of a coal liquefaction plant, employing a particular deashing technology for a given set of cost, pricing, financing and inflation assumptions using a 20 year return on invested capital on a discounted cash flow basis (DCF).

6.1.2. Model Description

For the purposes of this economic comparison, a generic coal liquefaction facility has been divided into a number of major unit operations, for which capital costs can be assigned based on throughputs as shown below:



For each of these plant sections, technical performance is estimated for a given plant configuration. On the basis of plant throughput, unit operations are sized and capital costs calculated. The model is sufficiently flexible to permit:

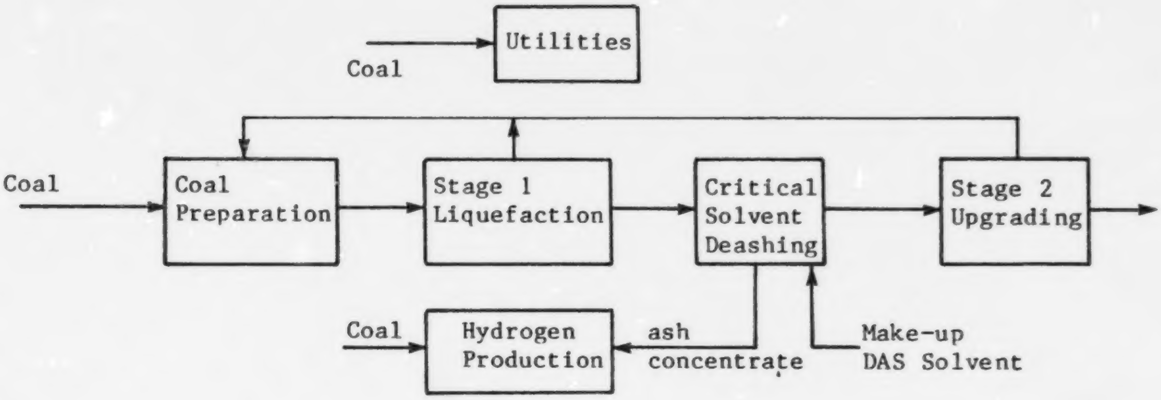
- variations in coal type and quality
- coal conversion efficiency
- recycle rate
- deashing performance
- level of upgrading of coal liquids.

In the model, hydrogen requirements are calculated stoiciometrically based on coal elemental analysis and final product mix. Hydrogen, can be generated from natural gas by steam reforming or by gasification of residuals plus coal. Although the model can calculate a cost/barrel of liquid product, its main utility is to determine the relative economics of various cases of the generic model and for assessing the sensitivity of a typical projects' financial return to changes in the model assumptions.

6.1.3. Cases for Comparison

In this study, two main cases were selected for study. The base case was a two-stage liquefaction plant with inter-stage deashing using the Critical Solvent Deashings Process (CSD). Hydrogen production is through gasification of residuals with coal as required. Figure 6.1 illustrates this case.

FIG. 6.1 CSD DEASHING CASE - CASE A


Coal
Utilities
Coal
Coal Preparation
Stage 1 Liquefaction
Critical Solvent Deashing
Stage 2 Upgrading
Coal
Hydrogen Production
ash concentrate
Make-up DAS Solvent

The Centrax Deashing Process can also be employed in an inter-stage model; however, as a result of greater recovery predicted for the asphaltenes and pre-asphaltenes, the Centrax case opens up the possibility for hydrogen production by reforming natural or process gases or by gasification of residuals plus coal.

FIG. 6.2 CENTRAX DEASHING CASE - HYDROGEN BY GASIFICATION - CASE B-1

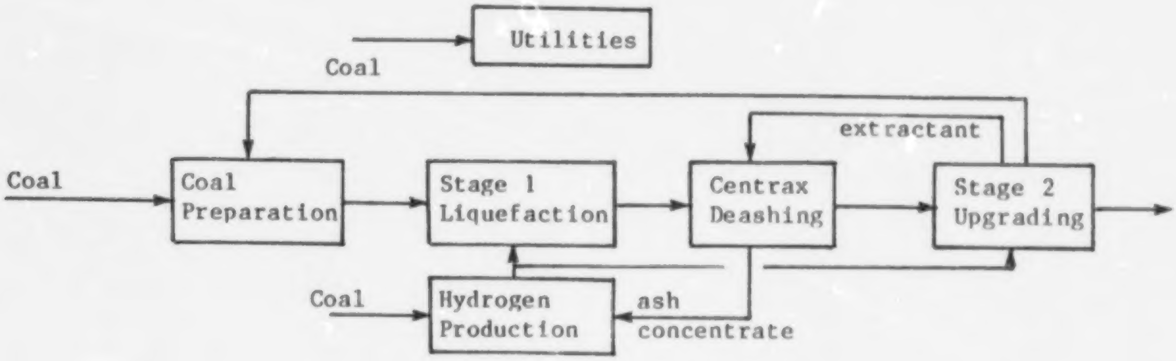
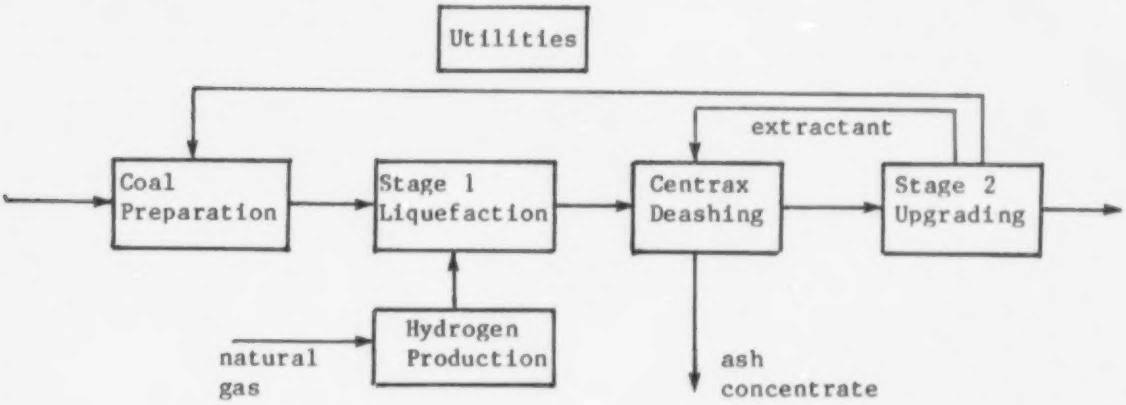

Utilities
Coal
extractant
Coal
Coal Preparation
Stage 1 Liquefaction
Centrax Deashing
Stage 2 Upgrading
Coal
Hydrogen Production
ash concentrate

FIG. 6.3 CENTRAX DEASHING CASE - HYDROGEN BY GAS REFORMING - CASE B-2


Utilities
extractant
Coal Preparation
Stage 1 Liquefaction
Centrax Deashing
Stage 2 Upgrading
natural gas
Hydrogen Production
ash concentrate

In the case of hydrogen generation by steam reforming of natural gas, it is assumed that the ash concentrate is used for power generation. However, the ash concentrate is given a residual value and power is purchased.

6.1.4 Model Documentation

the model has been developed using a LOTUS 1-2-3 spread sheet method which permits ready intereaction between model variables and assumption. Appendix 6 contains a sample output of the model for case B-1 and a printout of the formulas used in each of the cells of the spreadsheet.

6.2 Model Assumptions

6.2.1. Technical Assumptions

A number of technical assumptions have been made in the model for the purpose of comparing the relative economics of the cases on a reasonable and consistent basis. The model assures a final C_4+ production of 10,000 bbls/stream day of final product. Coal and hydrogen consumption are calculated depending on coal quality, liquefaction and upgrading conversion characteristics, and deashing efficiency. Listed below are the prime assumptions used in the model.

	CSD DEASHING	CENTRAX DEASHING	
	(Case A)	Gasification Case B-1	Gas Reforming Case B-2
C_4 - Production (bbls/sd)	10,000	10,000	10,000
Recycle Solvent (% new feed)	174	174	174
Deashing Factor (% solids in ash concentrate)	57.0	96.0	96.0
DAS Solvent Loss (% MAF Feed)	1.4	-	-
Hydrogen Production	Gasification	Gasification	Gas Reforming
Upgrading Case	High Yield & Low Yield	High Yield & Low Yield	High Yield & Low Yield

These cases have been compared using a typical Canadian lignite as coal feed. The lignite has 20.48% ash and 5.4% hydrogen (MAF basis).

The characteristics of this lignite on liquefaction and second stage upgrading is assumed to be as follows:

	Liquefaction Yield (% MAF Coal)	Upgrading Yield (% SRC Feed)	
		Low Yield	High Yield
H_2O	5.3	8.9	9.2
H_2S, SO_2	1.2	1.0	1.2
NH_3	0.3	1.9	2.1
CO, CO_2	1.0	-	-
C_1-C_5	5.9	4.6	8.9
Naphtha	4.3	11.2	20.3
Light Distillate 450°F	4.4	14.3	21.0
Distillate 450°F-FBP	23.0	25.0	32.0
SRC	48.9	37.8	11.4
Unreacted Coal	8.0	-	-

On the basis of these characteristics and the price assumptions the model has calculated values for a number of significant process requirements.

	CSD DEASHING	Centrax Deashing	
	(Case A)	Case B-1	Case B-2
Coal Use			
Liquefaction	3,206	2,438	2,438
Steam	164	157	-
Gasification	272	823	-
Total	3,643	3,418	
C_4 + Yield (bbls/MAF Tonne)	3.76	4.94	4.94
H_2 Consumption (% MAF Coal)	4.41	5.54	5.54

6.2.2 Operational Assumptions

It is assumed that the liquefaction facility in all cases will operate 92% of the time. This is an average over the life of the facility.

6.2.3 Pricing Assumptions

The pricing assumptions are very important to the model predictions. The relative cost of feed materials and final products are more critical than the absolute values. Although in recent months energy prices have collapsed, it must be remembered that this model is for a plant built currently with revenues over the period 1990 - 2010. For the purposes of this analysis, crude oil is used as a bench mark at $27.50 (U.S.) per bbl. products as valued relative to this benchwork.

	% of Benchmark
Naphtha	120
Light Distillate	110
Distillate	100
SRC	70

Other Feed materials are valued as follows:

Coal ($US/MF Tonne)	$ 35.00
Natural Gas ($US/1000 SCF)	$ 1.50
Purchased Power ($US/KWh)	$ 0.04
Residual Fuel ($US/M Btu)	$ 1.25
DAS Solvent ($US/bbl)	$ 42.00

All costs are assumed to increase at a rate of 8% per year. Over the 20 year project life, the value of the products will increase at a rate 1% greater than the average rate of inflation.

6.2.4 Capital Cost Assumptions

The capital cost of the liquefaction facility will be dependent on the plant size, unit operations used and the throughputs through each section of the plant. It is recognized that an exact cost for a coal liquefaction plant cannot be developed for each case; indeed, until plants of this size are actually committed to construction the cost is open to some debate. However, for the purpose of this study, typical values reported elsewhere for commercial liquefaction facilities should suffice. Table 6.1 lists the cost factors used for the 7 plant sections employed.

TABLE 6.1 CAPITAL COST FACTORS

Plant Section	Cost Basis	Cost Factor
Coal Preparation	Coal Feed (TPD)	\$ 5,300
Liquefaction	Coal - Recycle Solvent Throughput (TPD)	\$ 6,700
Deashing		
Centrax	Feed to Deashing (TPD)	\$ 9,200
or		
CSD	Feed to Deashing (TPD)	\$ 3,700
Upgrading	450° + material converted (TPD)	\$ 140,000
Hydrogen Production		
Gasification	Per each 1% of H_2 Consumption	\$ 4,400
or		
Reforming	Per each 1% of H_2 Consumption	\$ 2,290
Miscellaneous	Total Plant Feed (MF TPD)	\$ 12,100

On the basis of the cost factors shown in Table 6.1 a total plant capital cost is developed. In addition to this, further cost factors are employed.

Contingency	50%
Engineering	8%
Working Capital, Starting Capital and Financing during Construction	20%

Compounded, these factors increase the total plant investment by 94%. The capital cost for Centrax Deashing is estimated to be about 2.5 times that of CSD Deashing using conservative estimates of Centrax Separation Capacities.

6.2.5 Financing Assumptions

Since the approach taken to compare the cost benefit of difference coal liquefaction technoligies is a rate of return, the model requires certain project financing assumptions to be made. These are:

Equity Financing	60%
Debt Financing	40%
Interest Rate on Debt	13%
Amortization Period	20 years

6.3. Model Predictions

6.3.1. Economic Significance of Centrax Deashing

The model predicts Centrax inter-stage deashing to have real economic advantage compared to the less efficient CSD process. The following table presents the results of the economic analysis using the rate of return method.

Percent Internal Rate of Return (Before-Tax)

Level of Upgrading	CSD Deashing	Centrax Deashing	
		Gasification	Gas Reforming
Low Severity Upgrading	9.2	11.7	17.11
High Severity Upgrading	9.9	12.5	18.5

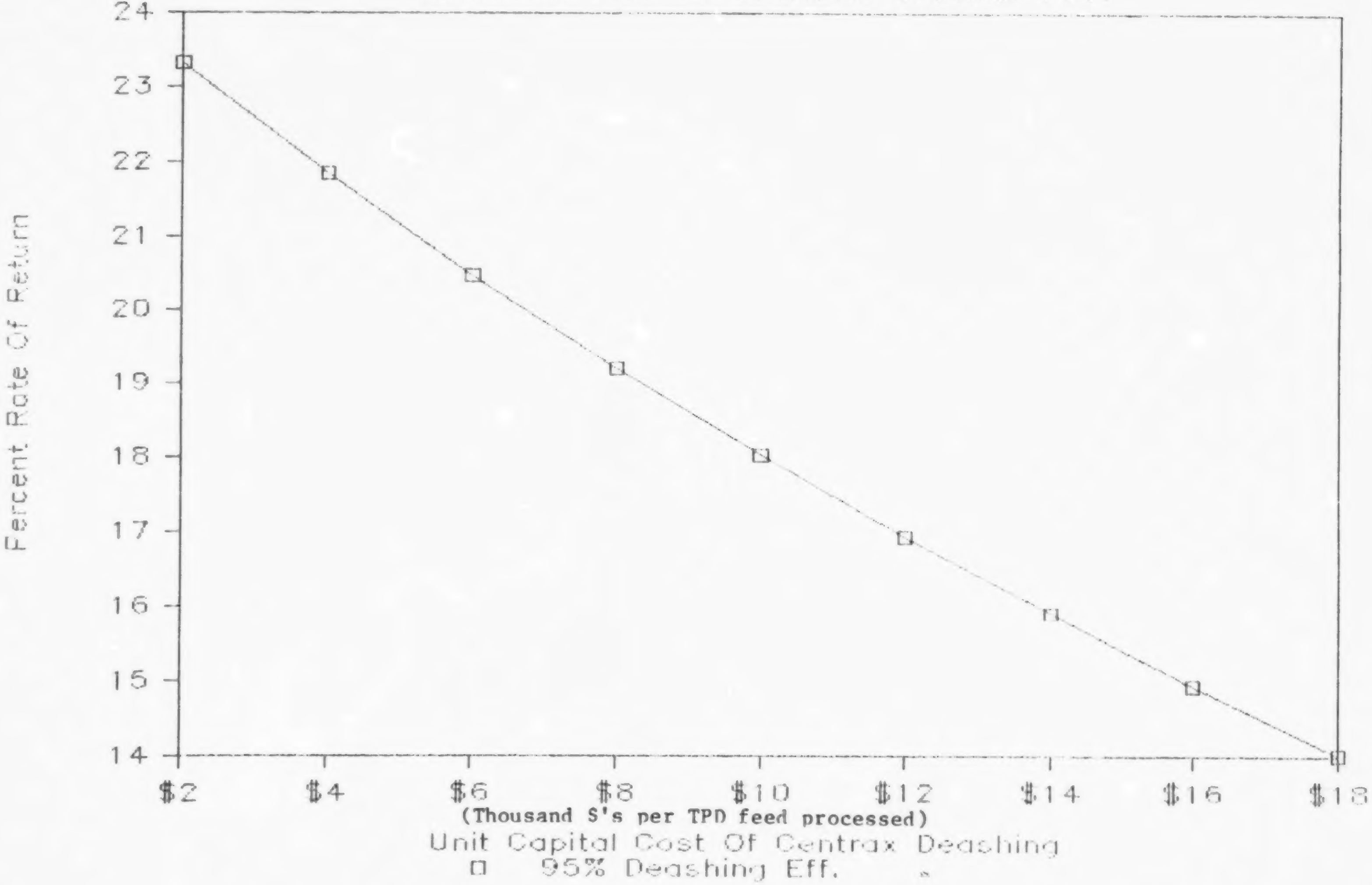
The economic benefits are the result of two main factors - the greater yield of liquids from the feed coal (4.61 vs 3.57 bbls distillate/tonne) and the cost advantage of using natural gas to produce hydrogen. While the advantage of using gas will depend on price and availibility to the project and is expected to vary widely, the Centrax deashing process still offers considerable advantage even when gasification is used to produce hydrogen. The reason for this is related to the small throughputs of coal to produce the same volume of coal liquids resulting in a lower operating costs for power and plant fuel. As well, the Centrax Process does not require any external make-up solvent.

6.3.2 Sensitivity to Capital Cost of Centrax Deashing

The capital cost of Centrax Deashing has yet to be determined pending on-going development of the Centrax Separator and establishment of capacity factors for first-stage liquefaction products. As a result, very conservative estimates of the capital cost of Centrax systems has been used. Figure 6.4 illustrates the effect on rate of return for the cost of Centrax Deashing over a very wide range. The comparison with the CSD process has been conducted at $9,200/daily tonne of deashing feed or about mid-point in the range selected for the sensitivity analysis. The figure shows that although the cost of deashing will effect the rate of return, the benefits of complete deashing are such that money spent to achieve high deashing factors is of real advantage to the overall project economics.

FIG. 6.4 SENSITIVITY ANALYSIS

ECON. RETURN vs. CENTRAX CAPITAL COST



7.0 DEVELOPMENT PLAN - PROTOTYPE TO EXTENDED TIME TRIALS

The development of Centrax technology, has been underway for about three years. The process has advanced to the extent that a multi-functional Centrax separation and extraction prototype unit has been designed and constructed with the capacity to handle flow up to approximately 5 L/min. The next stage of Centrax Development Program is to advance to fully continuous, multi-stage equipment in order to permit the demonstration of the Centrax process at a pilot plant scale such as at Wilsonville.

The Centrax deashing process is highly relevant to the objective of developing efficient and reliable alternative energy sources. The Centrax solution to the deashing problem has the potential to contribute significantly to coal liquefaction and coprocessing developments by:

- increasing net liquid yields
- making liquefaction more feasible for low-rank and high-ash coals
- opening opportunities for removing ash initially, inter-stage or as a final stage.

The Centrax process involves equipment for two basic process steps. In the first step the liquefaction products are recovered by displacement from the solids. This is accomplished through the use of a process-derived extractant applied in two or more stages in a cross-or counter-current mode while the solids are settled using centrifugal forces. This Centrax separation step requires uniquely developed equipment, of which a multi-functional single-stage unit has already been designed and fabricated. The second step involves the recovery of the volatile extractant from the compacted solids produced in the first step in a continuous liquid recovery unit (LRU). (See for example Figure 7.1).

The general objective of this phase of the Centrax Technology Development Program is to advance the development of the process and related equipment to the point that a pilot scale demonstration program could be undertaken at for instance the Wilsonville pilot plant for a limited period.

The specific objectives of this program are:

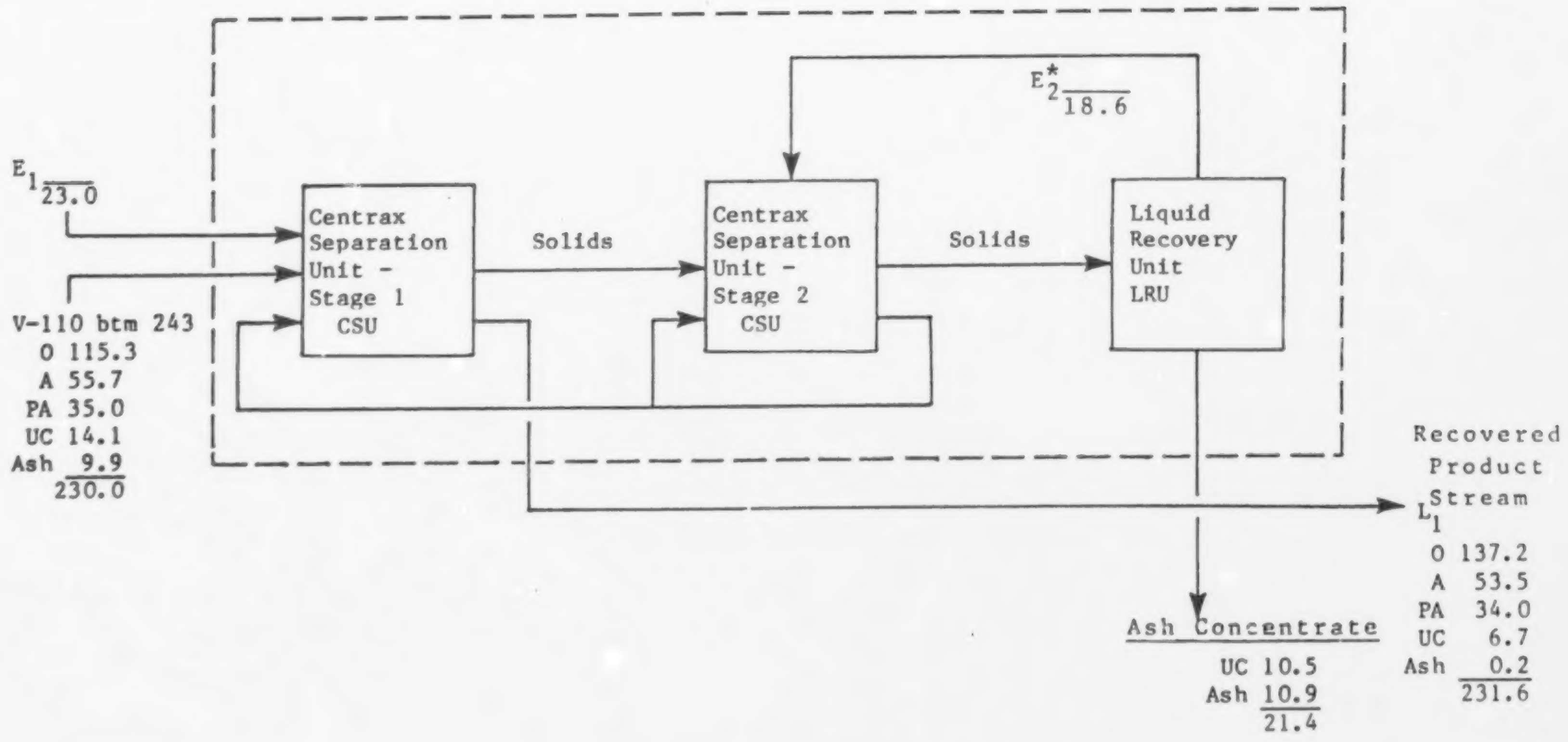
1) To test and evaluate the performance of the Centrax Separation Unit (CSU) Prototype One Unit leading to the design and construction of a multi-stage unit suitable for a Wilsonville scale operation - Prototype Two,

2) to scale-up batch and bench-scale extractant recovery apparatuses to a full continuous LRU device of a scale compatible with an integrated operation with the CSU,

3) to operate the CSU and LRU stages separately and together to develop process and equipment operating experience, including:

    - mass balance
    - energy balances
    - recovery efficiencies
    - problem solving in material handling
    - optimum processing conditions,

FIGURE 7.1 EXAMPLE OF CENTRAX FLOW DIAGRAM
PROCESS SCHEME 1 (WILSONVILLE RUN 243 FEED)



E_1 23.0
V-110 btm 243
O 115.3
A 55.7
PA 35.0
UC 14.1
Ash 9.9
230.0
Centrax Separation Unit - Stage 1 CSU
Solids
Centrax Separation Unit - Stage 2 CSU
Solids
Liquid Recovery Unit LRU
E_2^* 18.6
Recovered Product Stream L_1
O 137.2
A 53.5
PA 34.0
UC 6.7
Ash 0.2
231.6
Ash Concentrate
UC 10.5
Ash 10.9
21.4

NOTES

┌ ─ ┐ └ ─ ┘ Sandwell Centrax Process
* - streams obtained by evaporation
Units are scaled to 100 units of MF coal feed to the ITSL process
E - extractant

4) to generate quantities of Centrax deashing products for use in evaluating the impact of Centrax deashing on downstream and possibly upstream processing,

5) plan for an on-site demonstration program of Centrax technology at a large scale pilot plant of a size of for instance Wilsonville.

7.1 Scope of Work

The work to be performed relates to five tasks: Centrax Separation Unit development, Liquid Recovery Unit development, Integrated Centrax Separation and Liquid Recovery Operation, a study on the impact of Centrax deashing on coal liquefaction performance, and planning for an on-site demonstration of Centrax technology and equipment.

Figure 7.1 illustrates a typical flow sheet for the Centrax Deashing Process which was found to provide complete separation of liquids and solids using inter-stage liquefaction products from Wilsonville. This process requires three actual units to effect the solids separation and liquid recovery. The following table describes the purpose, status of development of these units and the goals of this proposal to advance the Centrax process.

TABLE 7.1 CENTRAX PROCESS EQUIPMENT DEVELOPMENT STATUS & GOALS

	Centrax Separation Unit (CSU)		Liquid Recovery Unit (LRU)
	Stage 1	Stage 2	
Function	Initial Solids Separation	Displacement of non-volatile liquids from cake	Recovery of liquids from cake
Development Status	Multi-functional Single-Stage Device to simulate multiple stages; prototype completed November 1984		Batch vacuum oven
Objective	Multi-Stage CSU Unit.		Continuous liquid recovery device to operate in tandem with CSU

As can be seen in Figure 7.1, the three stages of this particular Centrax process configuration are closely-coupled to minimize the handling of the solids and the processing time. This requires fully continuous operation with respect to the transfer of both liquids and solids between processing stages. In the description of the tasks to follow the development objectives of each of the units will be described.

Task 1 - Centrax Separation Unit (CSU) Development and Demonstration

A multi-functional Centrax Separation Unit which can be used to simulate the processing steps involved in both separation stages has been designed, fabricated and commissioned. This unit has been developed according to the requirements of the Centrax process. This initial CSU prototype which is called Prototype 1 has several key features which distinguishes it from centrifugal separators commercially available. These are:

- specific design features to operate in a multi-stage mode to achieve the displacement and recovery of interstitial liquids from non-filterable cakes,
- designed to process materials at elevated temperatures,
- designed to minimize problems with abrasive slurries,
- achieve separation of very small discrete particles,
- handle low and high solid feed concentrations,
- handle low and high non-distillate feed concentrations.

The overall focus of Table 7.1 is firming-up of the process and equipment requirements through testing of the CSU Prototype 1. Specific sub-tasks in Task 1 are as follows:

1.1 CSU Prototype 1 Performance Evaluation

A series of performance trials will be undertaken with the CSU Prototype Unit in order to evaluate the design features of the equipment and the extent to which the processing requirements are being met. One of the difficulties of developing centrifugal equipment is in scaling-down the size of equipment while maintaining the desired G-force range of operation. The Prototype 1 unit has been designed to operate with the minimum possible flow requirements (approximately 5 L/min) and yet large volumes will still be processed in relatively short periods. There are two probable options for the supply of feed materials to the PDU. These are:

1) Moving on-site to for instance a Wilsonville size pilot plant to have convenient access to larger process streams.
2) Processing large batch volumes of feed transported from such a pilot plant to local testing facilities, with re-constitution of products for subsequent re-processing.

The second option will undoubtedly be the least expensive and yet still provide sufficiently reliable feed materials such that equipment mechanical and process performance can be further evaluated at this early stage of equipment evaluation.

1.2 CSU Prototype 1 - Extended Operation Performance

Operation of the CSU for extended periods to establish mechanical reliability and process stability is necessary before progressing to more advanced prototypes. It is proposed therefore to run the unit continuously for a period up to 100 hours to assess if there are any problems in the hardware design concepts which may only arise in continuous operation. Of particular interest will be the confirmation of reliable movement of solids through the unit without build-up.

1.3 CSU Prototype 1 - Renovations and Upgrades

Any problems in the operation of Prototype 1 revealed in the tests and trials conducted in Task 1 will be the subject of intensive study and redesign to solve these problems. The CSU has been designed such that the modification to the parts of the unit in which the separation and extraction occurs can be achieved relatively quickly and cost effectively. Renovations and upgrades will be performed on the CSU quickly to solve the type of problem that only can be determined by actual process operation. It must be recognized that the modifications would be made concurrent with Tasks 1.1 and 1.2 as the need arises and not necessarily all at the end.

1.4 CSU Prototype 1 - Performance Trials and Process Optimization

Upon completion of the renovations and grades, as required to the prototype, another set of performance trials will be undertaken with new feed materials. The effectiveness of the design changes will be assessed in this further set of performance trials which will also have the purpose of updating the process performance in terms of:

- separated liquid quality
- cake compression
- asphaltene and pre-asphaltene extraction
- process conditions
    - temperatures
    - feed rates
    - cycle times
    - extractant/feed ratios

Sub-task 1.4 should complete Task 1 having advanced to the point of a properly functioning Prototype 1, the establishment of design criteria for the next prototype level and confirmation of the bench-scale data.

Task 2 - Liquid Recovery Unit (LRU) Development and Demonstration

The recovery of the volatile liquids from the cake exiting from for instance the second-stage CSU is an important part of the Centrax deashing process. Fractions such as asphaltenes and pre-asphaltenes have been displaced from the solids cake in the CSU stage of the process. This allows the solids fraction to be concentrated up to 100% solids in the ash concentrate by the recovery of the interstitial liquids in the solids by using their volatile properties. This task addresses the development of a prototype continuous liquid recovery unit (LRU).

2.1 LRU Prototype 1 - Design and Construction

Sandwell has conducted extensive bench-scale studies to define the appropriate conditions for rapid effective recovery of volatile materials from the ash concentrate. In this sub-task the known-how gained in this bench-scale study will be used to design a continuous unit to evaporate off and collect the residual liquids in the cake as well as discharge the final dry ash concentrate. The unit will have the following features.

- throughput capacity compatible with CSU prototype 1.
- continuous operation with respect to feed, liquid recovery and dry solids ejection
- flexibility in design operation conditions for a range of variables to be investigated

Upon completion of the design specification a prototype LRU will be fabricated, installed and commissioned.

2.2 LRU Performance Trials

The products generated in Task 1 with the CSU will be reserved for final separation in the prototype LRU. The purpose of these performance trials will be to adjust the processing conditions to give reliable and effective liquid recovery and to evaluate the operating performance of the prototype LRU. As required, the prototype LRU will be modified to improve operability and performance. Sub-task 2.2 should conclude with successful operation of the LRU stage.

Task 3 - Integrated CSU and LRU Demonstration of Centrax Deashing

The previous tasks will have advanced the status of the process and equipment development such that a full integrated demonstration can be performed. This demonstration should be considered as a prerequisite to an on-site demonstration at a selected facility. The final equipment set-up would also be the very same for an on-site demonstration.

3.1 Fabrication of Multi-Stage CSU

The operating experience gained on the CSU Prototype 1 will be used to design a multi-stage CSU having separate separation stages. This unit will be called CSU Prototype 2. While Prototype 1 was a multi-functional single-stage unit used to develop design and process information for the different stages, the Prototype 2 will have distinct stages which can be operated in tandem rather than alternately in the prototype 1 simulation. Furthermore, the CSU Prototype 2 may have different design and process conditions for the different stages. The units will however be closely-linked to effect inter-stage mixing and minimize solid handling.

3.2 CSU Prototype 2 - Commissioning and Performance Trials

The new CSU-2 will be commissioned and operated at the testing facilities. The performance trials will serve to study the effects of tandem operation of this latest prototype and further refine the process conditions for stages 1 and 2.

3.3. Integrated CSU-2 and LRU Operation

In a final task, the liquid recovery unit (LRU) designed, built and tested in Task 2 will be tested in conjunction with CSU Prototype 2. This will represent a complete continuous demonstration of the Centrax deashing process. It is planned to operate this series of tests using fresh materials obtained from a suitable large scale pilot plant, for example from Wilsonville. On the basis of these tests, a complete pro-forma mass balance will be developed to represent what an on-site operation could achieve. Extended operations would be conducted with re-constituted samples to study the performance and reliability over longer periods.

Task 4 - Investigation of Impact Upstream and Downstream Processing Related to the Introduction of Centrax Deashing

Preliminary bench-scale studies conducted with samples from Wilsonville Run 243 have indicated that the Centrax process prevents retrogressive reaction during inter-stage deashing while at the same time recovering more asphaltenes and pre-asphaltenes. It is therefore proposed to use some of the products separated by Centrax deashing to study the impacts on the upstream and downstream steps of a process.

Batch autoclave and continuous bench scale studies will be performed, over extended time periods, to determine if the material separated by Centrax has either beneficial or negative effects, as compared to material separated in other ways. The investigation will focus on fouling of catalysts, quality of product, and on product slate.

Task 5 - Process Engineering Studies

5.1 Process Optimization

Process engineering studies will be performed to address the following:

a) mass and energy balances of the process,

b) optimization of process conditions for Centrax deashing,

c) scale-up consideration for commercial scale.

5.2 On-site Demonstration Plans

Logistic plans will be developed for an on-site demonstration of the Centrax at a suitable plant or large scale pilot plant. Included in the planning will be consideration of

- timing,
- compatibility with the program of the selected site,
- space and operational needs,
- feed, services and support requirements.

7.2 Project Schedule

The proposed project will be completed in 12 months. A detailed schedule is shown in the attached Table 7.2.

7.3 Cost Estimate

The total cost for the work program describe in sub-section 7.1 is estimated to be $900,000 Cdn. This does not include any possible costs for the required amounts of feed slurry samples.

Table 7.3 provides a summary of the costs, and Table 7.4 presents a breakdown of costs by task for direct labour while Table 7.5 gives a similar cost breakdown of costs for disbursements. The total costs by task are summarized in Table 7.6.

Appendix 1

Pilot Plant Operating Conditions

Test Run Data Log Sheets

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TABLE 7.2    PROJECT SCHEDULE
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                                                                                    MONTHS
                                       ------------------------------------------------------------------------------------------------------------
TASK     DESCRIPTION                       1        2        3        4        5        6        7        8        9        10       11       12
======== ============================= ======== ======== ======== ======== ======== ======== ======== ======== ======== ======== ======== ========

Task 1   CENTPAK SEPARATION UNIT D&D
         1.1 CSU Performance Trials    ******************
         1.2 CSU Extended Operation                      *********
         1.3 CSU Modifications                  ***************************
         1.4 CSU Process Optimization                    ***************************

Task 2   LIQUID RECOVERY UNIT DEVELOP
         2.1 LRU Design & Construction ************************************
         2.2 LRU Performance Trials                               ******************

Task 3   INTEGRATED CSU & LRU OPERATION
         3.1 Design & Fab CSU - 2                                                   ************************************
         3.2 CSU - 2 Performance Trials                                                                                 *********
         3.3 Integrated CSU-2 & LRU Op.                                                                                          ******************

Task 4   INTEGRATED IMPACT STUDIES
         4.1 Batch Work                                                             ***************************
         4.2 Continuous Studies                                                     ***************************

Task 5   ON-SITE DEMONSTRATION PLANNING

         5.1 Process Engineering Study                                              *********************************************
         5.2 Demonstration Planning                                                                            ************************************

Task 6   REPORTING SCHEDULE                                    ###                        ###                        ###                     ######

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TABLE 7.3 COST SUMMARY TABLE

		TOTAL COST	
COST ITEM		$ CDN	$ US
TOTAL ESTIMATED LABOUR(AT COST)		$133,189	$101,224
OVERHEAD FACTOR	175.0%		
TOTAL OVERHEAD COSTS		$233,081	$177,142
TOTAL ESTIMATED DISBURSEMENTS (AT COST)		$463,000	$351,880
TOTAL ESTIMATED COST		$829,271	$630,246
FEE(% OF TOTAL)	7.5%	$62,195	$47,268
IN-KIND CONTRIBUTION USE OF MULTI-FUNCTIONAL PROTOTYPE		$8,145	$6,190
TOTAL PROJECT COST		$899,611	$683,704

Cost in US $'s estimated at exchange rate of $1.32 CDN $'s = 1.00 US $'s

TABLE 7.4 ESTIMATED LABOUR COST ($CDN)

TITLE: CENTRAX DEMONSTRATION
DATE: NOV 12,1984
ESTIMATED BY: SLH/PGG

TASKS	ESTIMATED LABOUR (Mandays) CATEGORY 810	820	830	840	850	860	870	880	900	TOTAL TIME (Manhours)	TOTAL COST ($'s)
Task 1 CENTRAX SEPARATION UNIT D&D											
1.1 CSU Performance Trials	2.0	10.0		5.0	20.0	5.0	10.0	5.0		427.5	$7,120.50
1.2 CSU Extended Operation	2.0	10.0		5.0	30.0	20.0	20.0	10.0		727.5	$11,501.36
1.3 CSU Modifications	5.0	10.0				20.0	10.0	10.0		412.5	$7,650.17
1.4 CSU Process Optimization	3.0	10.0		5.0	20.0	10.0	10.0	15.0	5.0	585.0	$9,074.31
Task 2 LIQUID RECOVERY UNIT DEVELOP											
2.1 LRU Design & Construction	2.0	10.0			30.0	20.0	20.0	10.0		690.0	$10,919.79
2.2 LRU Performance Trials	3.0	10.0		5.0	30.0	20.0	40.0	10.0	5.0	922.5	$14,074.94
Task 3 INTEGRATED CSU & LRU OPERATION											
3.1 Design & Fab CSU - 2	3.0	20.0			10.0	20.0	40.0	15.0	3.0	832.5	$14,001.83
3.2 CSU - 2 Performance Trials	3.0	10.0		5.0	20.0	20.0	40.0	15.0	3.0	870.0	$13,432.86
3.3 Integrated CSU-2 & LRU Op.	2.0	10.0		5.0	20.0	10.0	20.0	15.0	2.0	630.0	$9,757.31
Task 4 INTEGRATED IMPACT STUDIES											
4.1 Batch Work	3.0	20.0		20.0			10.0	10.0	3.0	495.0	$9,190.20
4.2 Continuous Studies	3.0	20.0		20.0			10.0	10.0	3.0	495.0	$9,190.20
Task 5 ON-SITE DEMONSTRATION PLANNING											
5.1 Process Engineering Study	10.0	20.0			10.0				5.0	337.5	$7,513.01
5.2 Demonstration Planning	10.0	20.0			10.0	10.0	5.0	5.0	5.0	487.5	$9,762.90
SUB-TOTALS	51.0	180.0	0.0	70.0	200.0	155.0	235.0	130.0	34.0	7912.5	$133,189.38

TABLE 7.5 ESTIMATED DISBURSEMENTS ($CDN)

DISBURSEMENTS

TASKS	TRAVEL& SUBSISTENCE	COMMUNICATIONS	EXPENDABLE SUPPLIES	PROTOTYPE EQUIPMENT	SUB-CONTRACTS	USER FEES DISBURSEMENTS
Task 1 CENTRAX SEPARATION UNIT D&D						
1.1 CSU Performance Trials			$1,500			$1,500
1.2 CSU Extended Operation			$1,500			$1,500
1.3 CSU Modifications		$1,000		$30,000	$40,000	
1.4 CSU Process Optimization	$1,000	$1,000	$1,500			$1,500
Task 2 LIQUID RECOVERY UNIT DEVELOP						
2.1 LRU Design & Construction		$500		$100,000	$50,000	
2.2 LRU Performance Trials	$1,000	$1,000	$2,000			
Task 3 INTEGRATED CSU & LRU OPERATION						
3.1 Design & Fab CSU - 2		$500		$150,000	$50,000	
3.2 CSU - 2 Performance Trials		$500	$1,500			
3.3 Integrated CSU-2 & LRU Op.	$1,000	$500	$1,500			
Task 4 INTEGRATED IMPACT STUDIES						
4.1 Batch Work	$500		$1,500		$2,000	
4.2 Continuous Studies	$500	$500	$1,500		$2,000	
Task 5 ON-SITE DEMONSTRATION PLANNING						
5.1 Process Engineering Studie	$1,000	$500			$5,000	
5.2 Demonstration Planning	$2,000	$2,000			$2,000	
SUB-TOTALS	$7,000	$8,000	$12,500	$280,000	$151,000	$4,500
TOTAL ESTIMATED DISBURSEMENTS (AT COST)						$463,000

TABLE 7.6 ESTIMATED COST BY TASK

TASK	DESCRIPTION	TOTAL COST BY SUB-TASK	TOTAL COST BY TASK ($ CDN)	TOTAL COST BY TASK ($ US)
Task 1	CENTRAX SEPARATION UNIT D&D			
	1.1 CSU Performance Trials	$24,275		
	1.2 CSU Extended Operation	$45,371		
	1.3 CSU Modifications	$98,941		
	1.4 CSU Process Optimization	$32,201		
			$200,788	$152,599
Task 2	LIQUID RECOVERY UNIT DEVELOP			
	2.1 LRU Design & Construction	$194,069		
	2.2 LRU Performance Trials	$45,909		
			$239,978	$182,383
Task 3	INTEGRATED CSU & LRU OPERATION			
	3.1 Design & Fab CSU - 2	$256,930		
	3.2 CSU - 2 Performance Trials	$41,861		
	3.3 Integrated CSU-2 & LRU Op.	$32,070		
			$330.861	$251,455
Task 4	INTEGRATED IMPACT STUDIES			
	4.1 Batch Work	$31,469		
	4.2 Continuous Studies	$32,006		
			$63,475	$48,241
Task 5	ON-SITE DEMONSTRATION PLANNING			
	5.1 Process Engineering Study	$29,198		
	5.2 Demonstration Planning	$35,312		
			$64,509	$49,027
		----------	----------	----------
			$899,611	$683,704

7.4 Program Implementation

Implementation of this 12 month program is estimated to cost $900,000 Cdn. It will progress the Centrax development from a prototype multi-functional single stage pilot plant separator, to a pilot plant consisting of:

- second generation separators,
- first continuous liquid recovery unit,
- integrated assembly made-up of several separators in series closely coupled to each other and the recovery unit,

ready for extended demonstration runs.

These runs may be an integral part of the operation of a large scale size fuels pilot plant complex, or they may be conducted on a side stream of such a facility.

Pilot Plant Operating Conditions

SUMMARY OF EXPERIMENTAL OPERATING CONDITIONS

RUN	DATE	TYPE OF FEED	FEED RATE	RPM	G FORCE	TEMPERATURE		AMOUNT PROCESSED
						FEED	SEPARATOR	
#			L/Min		Multiples of Gravity	°C	°C	Liters
10	JUL 25/85	Separated Liquid From Run 9	1.37 - 6.72	4600	2960	180	150	30.4
9	JUL 11/85	V110 247 - T105 1:0.3	1.20 - 7.27	4600	2960	180	110	29.7
			0.97 - 7.27	3800	2020	180	110	
8	JUN 24/85	V110 - T105 1:0.3	0.89	3800	2020	180	150	27.0
			0.89	4600	2960	180	150	
7	MAY 30/85	Recombined Mat. From Run 6	0.93	3800	2020	180	150	23.3
6	MAY 21/85	Recombined Mat. From Run 5	1.05	3800	2020	180	150	42.4
5	MAY 5/85	Recombined Mat. From Run 4	0.96	3700	1910	180	150	
4	APR 4/85	V110 - T105 1:0.1	0.93	4400	2710	180	-	44.4
3	MAR 7/85		1.11	4400	2710	180	-	28.0
2	FEB 19/85	Separated Liquid From Run 1	0.89	3600	1810	180	-	46.7
1	DEC 21/84	V110 - T105 1:0.4		2900	1180	180	-	

Test Run Data Log Sheets

RUN 1 5142 CENTRAX PILOT PLANT RAW DATA

RUN TIME	VESSEL 1		VESSEL 2		VESSEL 3	VESSEL 4	SEPARATOR			#	SAMPLES LOCATION	COMMENTS
	LEVEL	TEMP.	LEVEL	TEMP	TEMP.	TEMP.	SPEED	SLURRY TROUGH TEMP.	TANK TEMP.			
Min.	L.	C	L.	C	C	C	RPM	C	C			
-	84.4	178	12.7	180							Feed	
0:00	82.8	178		181	180	180	3000					Start Feed
2:00	78.3	178	11.6	180	180	180	3000					
5:00	73.2	178	9.4	180	180	170	3000					
8:00	65.3	178	8.4									
16:00	65.3	178	5.9	180	180	150	2900					
19:00	63.9	180	5.9	180	190	152	2900					
21:00	61.4	180	5.6	180	175	152	2900				Slurry	
28:00			4.7									
31:00	54.2	180	4.4	182	164	160	2900					
36:00											Product	
39:00	48.3	180	3.1	180	170	162	2900					
42:00	45.5	180	2.6	180	170	165	2900					
47:00	42.0	180	1.4	180	170	170	2900					
52:00											Slurry, Product	
53:00	37.1	180	0.7	180	173	170	2900					
56:00	31.3	180	0.4	180	178	170	2900					
58:00	28.8	180	0.3	180	178	170	2900					
61:00	25.0	180	0.2	180	178	170	2900					
66.00	20.6	180	0.0	180	180	170	2900					
72.00	12.6	180	0.0	180	180	170	2900					
76.00	9.1	180	0.0	180	182	170						
80.00	4.1	180	0.0	180							Slurry,product,feed	
89.00	1.2	180	0.0	180	183	170	2900					
94.00	0.0	180	0.0	176	188	170						Transfer slurry

```
CASE B-1: CENTRAL DEASHING - HYDROGEN BY GASIFICATION

************************    ******************************  ******************************************************
                                                                        TOTAL THRU(SIC)PUT   TOTAL AREA COST
     AREA/ITEM      UNITS              COST BASIS            FACTOR            TPD               ($s)
******************* ********  ************************** *********      ******************* ******************
COAL PREPARATION          1    COAL FEED(TPD)               $5,200                   3240       $17,188,933
LIQUEFACTION              1    ADJ. THRUPUT(TPD)            $6,700                   6711       $44,964,177
DEASHING-CENTRAL          1    DEASHING FEED(TPD)           $9,200                   5064       $46,584,890
        -CSD                   DEASHING FEED TPD            $[illegible]                0                $0
UPGRADING                 1    450C+ SMAF FEED CONV.       $14,[illegible]      [illegible]       $84,421,846
HYDROGEN-GASIFICATIO      1    SHI CONS MAF FEED            $4,4[illegible]         4.64%       $41,479,[illegible]
        -CH4                   SH2 CONS MAF FEED            $2,[illegible]    [illegible]                $0
MISCELLANEOUS             1    TOTAL PLANT FEED MF TPD)    $[illegible]              2450       $29,637,446
******************* ********  ************************** *********      ******************* ******************
SUB-TOTAL                                                                                     $264,279,065

CONTINGENCY                                                [illegible]                        $132,139,543
ENGINEERING & DESIGN                                       [illegible]                         $31,713,490
                                                                                          ==================

SUB-TOTAL                                                                                     $428,132,115
WORKING CAPITAL,START-UP,FEE,ETC                             20.00%                            $85,626,424
                                                                                          ==================
TOTAL CAPITAL INVESTMENT                                                                      $513,758,542

*****************************************************  ******************************************************


            Deashing Cost Factor ($/tpd feed)          $9,200

            Internal Rate of Return                     11.72%

            Deashing Factor                             70.00%

            Deashing Factor
                     $2                      $4      $6      $8             $10             $12
```

CASE B-1 CENTRAX DEASHING   HYDROGEN BY GASIFICATION

```
*********************************************************************************************************************************************************************************
       PRODUCT                  TOTAL    STAGE 1  DEASHING  STAGE 1   STAGE 2  STAGE 2  YIELDS    STAGE 2    TOTAL    SPECIFIC  DENSITY    YIELD        VALUE       REFERENCE       PROD. VALUE
    DISTRIBUTION                FEED     OUTPUT   RESIDUE  NET PROD.   FEED      LO       HI      PRODUCT   PRODUCT   GRAVITY  BBLS/TONNE BBLS/TONNE   FACTOR      OIL PRICE     DLRS/TONNE FEED
***************************  ********* ********* ********* ********* ********* ********* ********* ********* *********  ********* ********************* *********   ***********   ***************
H2O                                     5.30%     0.00%     5.30%     0.00%     8.90%     9.15%     4.25%     9.55%     1.00      6.29      0.60
H2S,SO2                                 1.20%     0.00%     1.20%     0.00%     0.95%     1.22%     0.47%     1.67%                                                                   $0.00
NH3                                     0.30%     0.00%     0.30%     0.00%     1.67%     2.08%     0.89%     1.19%                                                                   $0.00
CO,CO2                                  1.00%     0.00%     1.00%     0.00%     0.00%     0.00%     0.00%     1.00%                                                                   $0.00
C1-C5                                   5.90%     0.00%     5.90%     0.00%     4.57%     8.54%     2.12%     8.02%     0.78      8.06      0.65        78.40%        $27.50            $5.47
NAPHTHA                                 4.30%     0.00%     4.30%     0.00%    11.23%    21.03%     5.28%     9.58%     0.82      7.67      0.74       120.00%        $27.50           $24.44
LIGHT (450F                             4.40%     0.00%     4.40%     0.00%    14.75%    21.02%     6.84%    11.24%     0.90      6.99      0.79       110.00%        $27.50           $23.77
DIST SOLV (450F-EBP)                   23.00%     0.00%    23.00%     0.00%    24.95%    32.00%    11.90%    34.90%     0.95      6.62      2.31       100.00%        $27.50           $63.54
                                                                                0.00%               0.00%     0.00%     1.00      6.29      0.00       100.00%        $27.50            $0.00
SRC                                    48.90%     1.19%     0.00%    47.71%    37.84%    11.44%    18.05%    18.05%     1.05      5.99      1.08        70.00%        $27.50           $20.83
ASH                           20.48%   20.48%    20.48%                                                       0.00%
UNCONVERTED COAL                        8.00%     8.00%                                                       0.00%
                                                                                                              0.00%

H2 CONS                        2.90%                                  1.74%                                  -4.64%
  FEED COAL(MAF)             100.00%                                                                          0.00%
  FEED BITUMEN                 0.00%                                                                          0.00%
***************************  ********* ********* ********* ********* ********* *********           ********* *********  *********  ********************* *********   ***********   ***************
TOTAL NET PRODUCT(%MAF FEED)  120.48%  122.78%    29.67%    45.40%    47.71%   104.68%   106.13%    49.94%    95.34%                                                                 $138.05
RECYCLE (%MAF FEED)           173.97%                                173.97%    50.00%              50.00%
TOTAL THROUGHPUT              294.45%                                221.67%
STAGE 2  SRC CONVERSION(%MAF FEED)                                                                  29.66%
% SRC CONVERSION IN STAGE 2 FEED                                                                    62.16%
TOTAL DIST. PRODUCTION                                                                                                                  3.84                                            $111.76
C4+ PRODUCT VALUE                                                                                                                       4.92                                            $132.57
REFERENCE OIL PRICE                    $27.50
*********************************************************************************************************************************************************************************

                   HYDROGEN CONSUMPTION CALCULATION
                   **************************************************** ***************** ***************************** **********************************
                                   STAGE 1 FEED                         STAGE 1 H2 OUTPUT  STAGE 2 H2 INPUT              STAGE 2 H2 OUTPUT
                   ********************************************************************** ***************************** **********************************
                   FRACTION        STAGE 1   HYDROGEN  STAGE 1   STAGE 1   STAGE 1  ST2 FEED  HYDROGEN   STAGE 2   HYDROGEN  CONTENT        STAGE 2
                                   FEED      CONTENT   H INPUT   PRODUCT   H OUTPUT  (%MAF)   CONTENT    H INPUT     LO        HI            OUTPUT
                   ****************** ********* ********* ********* ********* ********* ********* *********  *********  ********* *********     *********
                   H2O                          11.11%     0.00%     5.30%     0.59%     0.00%    11.11%     0.00%    11.11%    11.11%          0.47%
                   H2S,SO2                       5.88%     0.00%     1.20%     0.07%     0.00%     5.88%     0.00%     5.88%     5.88%          0.03%
                   NH3                          17.65%     0.00%     0.30%     0.05%     0.00%    17.65%     0.00%    17.65%    17.65%          0.16%
                   CO,CO2                        0.00%     0.00%     1.00%     0.00%     0.00%     0.00%     0.00%     0.00%     0.00%          0.00%
                   C1-C5                        17.00%     0.00%     5.90%     1.00%     0.00%    17.00%     0.00%    17.00%    19.00%          0.37%
                   NAPHTHA                      11.90%     0.00%     4.30%     0.51%     0.00%    11.90%     0.00%    11.90%    12.50%          0.64%
                   LIGHT (450F                  10.90%     0.00%     4.40%     0.48%     0.00%    10.90%     0.00%    11.00%    11.50%          0.75%
                   DIST SOLV (450F-EBP)          8.60%     0.00%    23.00%     1.98%     0.00%     8.60%     0.00%     9.00%     9.60%          1.07%
                                                           0.00%     0.00%     0.00%     0.00%     0.00%     0.00%                              0.00%
                   SRC                           6.50%     0.00%    48.90%     3.18%    47.71%     6.50%     3.10%     7.50%     7.50%          1.35%
                   UNCONVERTED COAL              5.40%     0.00%     8.00%     0.43%     0.00%     5.40%     0.00%                              0.00%
                   FEED COAL         100.00%     0.054     5.40%     0.00%     0.00%     0.00%     5.40%     0.00%     5.40%     0.00%          0.00%
                   FEED BITUMEN        0.00%    10.70%     0.00%     0.00%     0.00%     0.00%    10.70%     0.00%    10.70%     0.00%          0.00%

                   TOTAL                                   5.40%               8.30%                         3.10%                              4.84%
                   ****************** ********* ********* ********* ********* ********* ********* *********  *********  ********* *********     *********
                   HYDROGEN CONS(%MAF FEED)                                    2.90%                                                            1.74%
```

SANDWELL TECHNOLOGIES

CASE B-1 CENTRAL REFINING HYDROGEN BY GASIFICATION

FINANCIAL MODEL (BEFORE TAX)

24-Feb

PROJECTED EARNINGS YEARS	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
PRODUCT REVENUES (MILLION U.S.$)	94.2	102.7	112.0	122.1	133.0	145.0	158.1	172.3	187.8	204.7	223.1	243.2	265.1	288.9	314.9	343.3	374.2	407.8	444.5	484.6
EXPENSES																				
CAPITAL CHARGES	29.3	29.3	29.3	29.3	29.3	29.3	29.3	29.3	29.3	29.3	29.3	29.3	29.3	29.3	29.3	29.3	29.3	29.3	29.3	29.3
COAL	38.1	41.2	44.5	48.0	51.9	56.0	60.5	65.3	70.6	76.2	82.3	88.9	96.0	103.7	112.0	120.9	130.6	141.0	152.3	164.5
BITUMEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
OTHER																				
NATURAL GAS	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
POWER	5.5	5.9	6.4	6.9	7.4	8.0	8.7	9.4	10.1	10.9	11.8	12.7	13.8	14.9	16.0	17.3	18.7	20.2	21.8	23.6
O&M	15.8	17.1	18.5	20.0	21.6	23.3	25.2	27.2	29.3	31.7	34.2	37.0	39.9	43.1	46.6	50.3	54.3	58.7	63.4	68.4
IN PLANT FUEL	3.7	4.0	4.3	4.7	5.0	5.4	5.9	6.3	6.8	7.4	8.0	8.6	9.3	10.1	10.9	11.7	12.7	13.7	14.8	16.0
CHEM.&CATALYSTS	4.0	4.3	4.7	5.1	5.5	6.1	6.6	7.2	7.9	8.6	9.4	10.2	11.1	12.1	13.2	14.4	15.7	17.1	18.7	20.4
RESIDUALS(CREDIT)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
BY-PRODUCT (CREDIT)	-2.9	-3.2	-3.5	-3.8	-4.1	-4.5	-4.9	-5.3	-5.8	-6.3	-6.9	-7.5	-8.2	-8.9	-9.7	-10.6	-11.5	-12.6	-13.7	-14.9
TOTAL EXPENSES	93.4	98.6	104.2	110.2	116.6	123.6	131.2	139.4	148.2	157.8	168.1	179.2	191.2	204.2	218.2	233.4	249.7	267.4	286.5	307.2
NET EARNINGS	0.8	4.1	7.8	11.9	16.4	21.4	26.8	32.9	39.6	46.9	55.1	64.0	73.9	84.7	96.7	109.9	124.4	140.4	158.0	177.4
																		NPV OF OTHER EXPENSES		
EQUITY	308.3																			
NET CASH	-307.5	4.1	7.5	11.9	16.4	21.4	26.8	32.9	39.6	46.9	55.1	64.0	73.9	84.7	96.7	109.9	124.4	140.4	158.0	177.4
CAPITAL COST/DAILY BBL	$42,813																			
INTERNAL RATE OF RETURN	11.72%																			
NET P V @8%	144.5																			

```
CASE B-1  CENTRAL DEASHING  - HYDROGEN BY GASIFICATION

 SUMMARY OF ASSUMPTIONS USED IN MODEL
                                                              HIGH SEVERITY UPGR.
 ***************************************************************************************************************************
 TECHNICAL                                   PRICING                          INFLATION                    FINANCING
 ASSUMPTIONS                               ASSUMPTIONS                      ASSUMPTIONS                  ASSUMPTIONS
 ********************************    ******************************    *************************    ******************************

 C4+ PRODUCTION(BBLS SD)     10000    CRUDE OIL BENCHMARK(US$/BBL  $27.50   OVERALL RATE(% ANNUM   8.00%   TOTAL INVESTMENT           $513.3
 COAL USE(MF TPD)                                                                                    (MILLION US $ $)
   LIQUEFACTION               2451    DISTILLATE PRODUCT           $28.12   RELATIVE RATES                 DEBT FINANCING(% OF TOTAL)  60.00%
   STEAM                       132    NON-DIST. PRODUCT            $19.25     PRODUCTS/BITUMEN     1.00%
   GASIFICATION                662                                          GAS                  0.00%   INTEREST RATE(% ANNUM)      12.00%
   TOTAL                      3245    COAL(US$ PER TONNE)          $25.00     COAL                 0.00%
 ASH CONTENT                17.00%                                          C4+                  0.00%   AMORTIZATION PERIOD(YRS)    20.00
 COAL USE(MAF TPD) LIQ        2037    NATURAL GAS(US$/1000 SCF      $1.56     POWER                0.00%
 BITUMEN/COAL RATIO            0.0                                          RESIDUALS            0.00%   ANNUAL CAPITAL CHARGE(%)    14.24%
 BITUMEN USE(MAF TPD)            0    PURCHASED POWER US$/KWH       $0.04
 FEEDSTOCK(MAF TPD)           2037                                                                     EQUITY INVESTMENT          $205.3
 %COAL IN SLURRY FEED        0.365
 RECYCLE FACTOR(%NEW FEED  173.97%    RESIDUALS(US$/M BTU)          $1.25                                (MILLION US $ $)
 DISTIL.YIELD(BBLS/MAF T)     3.34
 C4+ YIELD (BBLS/MAF T)       4.92    BITUMEN(US$/BBL)             $22.50
  H2 CONS (%MAF FEED)        4.64%
 COAL CONVERSION(%MAF FEE   92.00%    DAS SOLVENT (US$/BBL)        $42.00
 DEASHING FACTOR
   (%SOLIDS CONC.)          96.05%
 OPERATING FACTOR           90.00%

 POWER USE(KWH/T)              290
 GAS LOSS (%MAF FEED)        1.40%
 CONV.SEV(LO=0,HI=1)             0
 ***************************************************************************************************************************
```

Appendix 2

Analysis of Samples

CONFIDENTIAL

RUN 2 5142 CENTRAX PILOT PLANT RAW DATA

RUN TIME	VESSEL 1 LEVEL	VESSEL 1 TEMP.	VESSEL 2 LEVEL	VESSEL 2 TEMP	SEPARATION SPEED	SEPARATION SLURRY TROUGH TEMP.	SEPARATION TANK TEMP.	BEARINGS TEMP. TOP	BEARINGS TEMP. BOT.	#	SAMPLES LOCATION	COMMENTS
Min.	L.	C	L.	C	RPM	C	C	C	C			
-	25.4	180	47.3	168		176/126						
-	17.4											8 liters transfered in trough
0:00	17.4	180	48.2		3600							Start feed
7:00										1	Feed	
14:00	17.4		47.0	180								
17:00			44.8									
18:00			43.5		3000							
19:00			41.8									
20:00			40.4									
22:00			38.9									
23:00			37.6									
25:00			36.2									
26:00			35.0									
28:00			33.9									
29:00			32.6									
31:00			31.8									
32:00			30.6	180	3000	166/120						
36:00			24.8		3000	167/120						
37:00			23.9							2	Feed	
43:00			21.3							3	Separated Solid	
46:00			18.5	180	3000	170/120				4	Product	
51:00			12.7	182	3000	171/122						
56:00			9.8	182	3000	172/122						
59:00			5.1	181	3000	173/122						
63:00			1.7	182	3000	173/124						Stop feed

RUN 3 5142 CENTRAX PILOT PLANT RAW DATA

RUN TIME	VESSEL 1 LEVEL	VESSEL 1 TEMP.	FLOW	VESSEL 3 LEVEL	VESSEL 3 TEMP	SEPARATION SPEED	SEPARATION SLURRY TROUGH TEMP.	SEPARATION TANK TEMP.	BEARINGS TEMP. TOP	BEARINGS TEMP. BOT.	#	SAMPLES LOCATION	COMMENTS
Min.	L.		L/h	L.	C	RPM	C	C	C	C			
-	29.1	178			150		180/134						
0:00	28.7	178	60.0		152	4400	180/134						Start Feed
5:00											1	Product	
7:00	21.2	178	67.3		142	4400	180/135				2	Product	
12:00	15.2	178	62.0		150	4400	180/135				3	Feed	
13:00	14.8	178				2200							Speed Lowered
15:00	13.0		56.8		150	4400	180/135						Increased Speed
19:00		178				3000							Lower Speed
21:00						4400					4	Feed	Increased Speed
22:00	3.7		64		150	4400	178/136				5	Product	
25:00	0.7										6	Product	
26:00	0.0	178				4400	180/136						Stop feed

RUN 4 5142 CENTRAX PILOT PLANT RAW DATA

RUN TIME	VESSEL 1		FLOW	VESSEL 2		VESSEL 3 TEMP.	VESSEL 4 TEMP.	SEPARATION SPEED	SLURRY TROUGH TEMP.	BEARINGS TEMP.		#	SAMPLES LOCATION	COMMENTS
	LEVEL	TEMP.		LEVEL	TEMP					TOP	BOT			
Min.	L.		L/h	L.	C	C	C	RPM	C	C	C			
-	44.4	180		20.0		180	180		232/174	30	24			Transfer 4 liters into trough
0:00	44.3		72	16.4	180	180	180	4400	232/157	30	24	1	Feed	
11:00	34.0	180										2	Product	
18:00	27.3	180	180	16.4	180	175	180	4400	252/167	55	80			
20:00	25.6	180	60	16.4	180	178	180	4400	252/160	58	82			
27:00	19.1	180	60	16.4	180	178	180	4400	252/162	60	80	3	Product	
30:00	15.7		60											
34:00	12.6	180	60	16.4	180	180	180	4400	256/165	60	80	4	Product	
35:00														
37:00	7.2	180	60	16.4	180	180	176	4400	258/167	70	80	5	Feed	
40:00	5.5		30			180	178							
43:00	4.7	182	30		180			4400	254/167	80	90	6	Product	
	0.0													

RUN 5 5142 CENTRAX PILOT PLANT RAW DATA

RUN TIME	VESSEL 1		VESSEL 3		SEPARATOR			BEARINGS TEMP.		#	SAMPLES LOCATION	COMMENTS
	LEVEL	TEMP.	LEVEL	TEMP	SPEED	SLURRY TROUGH TEMP.	TANK TEMP.	TOP.	BOT.			
Min.	L.	C	L.	C	RPM	C	C	C	C			
-										1	Feed	
0:00	33.8	178			3700	150	200	55	45			Start feed
1:00	32.7	178										
3:00	31.4	178										No material collected
4:00	29.7	178			3700	150	200	55	48	2	Product	
6:00	28.0	178										
7:00	26.8	178										High alarm on feed
10:00	25.3	180			3700	150	200	58	50			Feed stopped
17:00	25.3	180										
36:00	31.8	178			2200	150	200	48	48			
39:00	31.9	178			3700	150	200	46	48			Start feed
42:00	29.5	178										
44:00	28.0	178										
47:00	23.5	180			3700	155	200	46	54	2	Product	
50:00	21.5	180								3	Product	
53:00	18.6	180										
54:00	17.0	180			3700	155	200	46	65	4	Product	
59:00	14.0	180										
60:00	12.0	180								5	Feed	
62.00	9.3	180								6	Product	
66.00	6.4	180										
68.00										8	Product	
71.00	1.5				3700	155	190	48	76			
72.00	0.3	180										
74.00	0.0				3700	155	190	48	82			Feed stopped
EOR										9	Vessel 3	
EOR										10	Vessel 3	
EOR										11	Feed	

RUN 6 5142

CENTRAX PILOT PLANT

RAW DATA

RUN TIME	VESSEL 1			VESSEL 3		VESSEL 4	SEPARATOR							
	LEVEL	TEMP.	FLOW	LEVEL	TEMP	TEMP.	SPEED	SLURRY TROUGH TEMP.	TANK TEMP.	BEARINGS TEMP. TOP	BEARINGS TEMP. BOT.	#	SAMPLES LOCATION	COMMENTS
Min.	L.	C	L/h	L.	C	C	RPM	C	C	C	C			
-	38.9	180		0.0	195	172		138	140	40	30			
0:00	39.1						3800			50	50			Start feed
2:00	35.4	180		4.1	180	174	3800	144	145	50	50	1	Feed	
5:00	33.2													
7:00	31.1			5.3										
9:00	29.2	180										2	Product	
11:00	27.7	180		7.8		180	3800	150	150	40	60	3	Feed	
13:00	24.9			9.4										
15:00	23.3			10.5								4	Product	
17:00	21.0	180		12.5		174	3800	154	150	40	68			
19:00	18.6			14.5								5	Product	
21:00	16.2			16.9								6	Product	
23:00	15.0	180	60	18.6	183	172	3800	160	153	46	72			Switch to Flow meter
26:00	12.3			20.2								7	Product	
28:00	10.5			21.7	180							8	Feed	
30:00	9.0			23.0	180	174	3800	162	155	50	74			
31:00												9	Product	
33:00	6.3			25.7	180	172	3800	162	155	50	78			
35:00	4.4			27.6								10	Product	
36:00	2.2	180		28.6			3800	162	155	50	78	11	Feed	
39:00	0.0			31.5										
EOR				33.08										transfer slurry from
EOR												12	Vessel 4	to Vessel 4
EOR												13	Vessel 3	

RUN 7 5142 CENTRAX PILOT PLANT RAW DATA

RUN TIME	VESSEL 1 LEVEL	VESSEL 1 TEMP.	VESSEL 3 LEVEL	VESSEL 3 TEMP	SEPARATION SPEED	SEPARATION SLURRY TROUGH TEMP.	SEPARATION TANK TEMP.	BEARINGS TEMP. TOP	BEARINGS TEMP. BOT.	#	SAMPLES LOCATION	COMMENTS
Min.	L.	C	L.	C	RPM	C	C	C	C			
-										1	Vessel 1	
0:00	38.4	180	0.0	180	3800	155	158	32	30			
3:00	34.7	180	1.1	175	3800	155	162	38	56	2	Product	
6:00										3	Product	Stop feed
7:00	34.7	180	2.1	178	3800	152	165	38	61			Start feed
8:00										4	Product	
11:00	30.7	180	5.0	178	3800	152	168	40	63			
12:00										5	Product	
14:00	27.5	180	7.4	178	3800	152	170	40	66			
15:00										6	Product	
17:00	24.1	180	9.0	174		152	174	45	72			
18:00										7	Feed	
19:00										8	Product	Scraper up
22:00	20.3	180	12.4	180	3800	152	176	48	74			
24:00												Scraper up
25:00	17.8	182	14.2	182			181	52	76	9	Product	
27:00	15.5		16.1					58	80			Stop feed
29:00					0	152	181	60	90			
30:00										10	Vessel 1	
30:00										11	Vessel 3	

RUN 8 5142 CENTRAX PILOT PLANT RAW DATA

RUN TIME	VESSEL 1 LEVEL	VESSEL 1 TEMP.	VESSEL 3 LEVEL	VESSEL 3 TEMP	SEPARATION SPEED	SEPARATION SLURRY TROUGH TEMP.	SEPARATION TANK TEMP.	BEARINGS TEMP. TOP	BEARINGS TEMP. BOT.	#	SAMPLES LOCATION	COMMENTS
Min.	L.	C	L.	C	RPM	C	C	C	C			
-						135/180	155	40	30			Start Feed
0:0	55.4	182	0.0	150						1	Product	
2:00	53.8	182	0.0	150	3800							
4:00	51.5	182	1.5	150	3800	120/180	152	40	32			
6:00	49.4											
8:00	47.4	182	4.1		3800	120/180	152	40	32			
9:00	46.3		4.5							2	Product	
10:40	44.8		5.3									
12:00	43.9	182	5.7	158				42	34			
12:40	42.9		6.2							3	Product	
15:00	41.1	182	7.2	158	3800	130/180						
16:00	40.0		7.6		4600							
17:00	39.2		8.0		4600					4	Product	
17:35	-		9.0		4600					5	Product	
19:30	37.0	182	9.6	158	4600	135/182	147	48	44			
20:20	36.0	180	10.6	158	4600	135/182	147	48	44			
23:30	34.3		10.8							6	Product	
24:00	33.5		11.6	158	4600							
25:00	32.2	180	11.9							7	Product	
26:00	31.4		12.3	158	4600	140/182	147	50	50			
27:00	30.6									8	Vessel 1	
28:00	29.1									9	Product	
29:20	28.4	180	13.4		4600	140/180		50	50			Stop feed
EOR										10	Vessel 3	

RUN 9 Page 1 5142 CENTRAX PILOT PLANT RAW DATA

RUN TIME	VESSEL 1 LEVEL	VESSEL 1 TEMP.	VESSEL 3 LEVEL	VESSEL 3 TEMP	SEPARATION SPEED	SEPARATION SLURRY TROUGH TEMP.	SEPARATION TANK TEMP.	BEARINGS TEMP. TOP	BEARINGS TEMP. BOT.	#	SAMPLES LOCATION	COMMENTS
Min.	L.	C	L.	C	RPM	C	C	C	C			
-					4600	110	135	32	28			Start Feed
0:00	29.4	180	0	180						1	Product	
1:40	27.4									2	Product	
2:30	26.4	180										Stop Feed
3:00	26.3	180	04	178	4600	110	135	32	30	3	Product	Scraper up decrease speed scraper up
5:00												
6:30	26.3				3800							
7:20	25.8				3800					4	Product	
8:20	24.7	180	1.4		3800	110	135	34	32	5	Product	
9:40	23.5	180	2.2		3800							Stop feed
10:12	23.2		2.5									
12:00			3.5		3800	110	137	35	32			
13:00										6	Product	Scraper Up
14:20	23.0		4.2		4600							Start feed
15:00	21.3		5.2							7	Product	Stop feed
18:20										8	Product	Scraper up
19:40					3800							
20:20	20.8									9	Product	Start feed
21:00	18.5	180			3800	120	140	44	36			Stop feed
24:16										10	Product	Scraper up
24:50					4600							
25:30	18.6											Start feed
25:48	16.3	180	8.3		4600	122	137	44	36	13	Product	Stop feed
28:49					4600					11	Product	Scraper Up
29:30	16.5		8.9									
31:15					3800							
31:45	16.4											Start feed
32:03	14.2		11.1	180	3800	120	132	46	40	12	Product	Stop feed
35:04					3800					14	Product	Scraper Up
37:00	14.2	180	11.1		3800							Automatic Mode
38:00	12.0											
38:40	10.9		13.8		3800					15	Feed	
39:20		180	14.4									Scraper Up
39:40	9.1											
40:40	8.7									16	Product	
41:40	7.4									17	Product	Scraper Up
42:30	6.8		15.3									

RUN 9 Page 2 5142

CENTRAX PILOT PLANT

RAW DATA

RUN TIME	VESSEL 1 LEVEL	VESSEL 1 TEMP.	VESSEL 3 LEVEL	VESSEL 3 TEMP	SEPARATION SPEED	SEPARATION SLURRY TROUGH TEMP.	SEPARATION TANK TEMP.	BEARINGS TEMP. TOP	BEARINGS TEMP. BOT.	# SAMPLES LOCATION	COMMENTS
Min.	L.	C	L.	C	RPM	C	C	C	C		
43.20	6.0		16.0							18 Product	
44:00	5.0		16.7		3800	125	130	46	42		Scraper up
46:00	2.4		18.0							19 Product	
46:20	1.9		18.3								
46:30	1.4		18.7								Scraper up
47:20	0.08		19.2		3900					20 Product	
47:40			20.3								Out of auto mode stop feed
49:25	0.07		21.0								
51:10	0.07				3900	125	130	44	42		
EOR	0.07	180	22.9							21 Vessel 3	

RUN 10 5142 CENTRAX PILOT PLANT RAW DATA

RUN TIME	VESSEL 1 LEVEL	VESSEL 1 TEMP.	VESSEL 3 LEVEL	VESSEL 3 TEMP	SEPARATION SPEED	SEPARATION SLURRY TROUGH TEMP.	SEPARATION TANK TEMP.	BEARINGS TEMP. TOP	BEARINGS TEMP. BOT.	#	SAMPLES LOCATION	COMMENTS
Min.	L.	C	L.	C	RPM	C	C	C	C			
-	43.7	182	0.00	172	-	150	165	40	30			
0:00					4600							Start feed
0:25										1	Product	
0:40	39.5		1.98		4600	140	165	52	32	2	Product	Stop feed
2:43										3	Product	Raise scraper
6:12	39.5		2.31		4600			53	48			Start feed
6:20										4	Product	
6:50										5	Product	
7:02	34.7		6.15		4600							
7:47										6	Product	Scraper up
9:33	34.6		6.45		4600	145	165	56	50	7	Product	Start feed
10:25	31.0		8.36							8	Product	
10:33	30.7		9.42									Stop feed
11:20						145	165	54	54	9	Product	Scraper up
13:08	30.7		9.72									Start feed
13:15										10	Product	
14:00	28.5									11	Product	
14:40	27.2	182	11.44					53	63			Stop feed
15:15										12	Product	Scraper up
17:50	27.3		11.64									Start feed
18:35	25.8		12.30		4600					13	Product	
20:15	22.7		14.68		4600			54	68	14	Product	
20:55	21.6		15.93									Stop feed
21:30										15	Product	Scraper up
23:25	21.5		16.53		4600			54	72			Start feed
25:00			18.32		4600			54	74	16	Product	
27:15	16.0		21.56		4600			54	74	17	Product	
29:15	13.3											Stop feed
30:00										18	Vessel 1	Stop Centrax
EOR										19	Vessel 3	
EOR												

Appendix 3

Results of Simultaneous Laboratory Separations

CONFIDENTIAL

ANALYSIS OF SAMPLES RUN 1

SAMPLE #	TIME Min from Start of Feed	TYPE/LOCATION	COMPOSITION				
			OIL	ASPHALTENES	PRE-ASPHALTENES	UNCONV. COAL	ASH
1	36:00	Separated Liquid				1.02	1.27
2	51:00	Separated Liquid				0.38	0.33
3	84:00	Separated Liquid				1.72	1.79
4	27:00	Redispersed Solids				1.58	0.23
5	51:00	Redispersed Solids				1.79	2.31
6	84:00	Redispersed Solids				4.08	4.92
7	BR*	Feed				2.47	3.03
8	E O R**	Redispersed Solids - in vessel				2.43	2.84
9	E O R	Separated Liquid - in vessel				0.69	0.62
10.	E O R	Sep. Solids - Under Solids Deflector				22.00	34.30
11.	E O R	Sep. Solids - Bowl Wall				11.81	15.53
12.	E O R	Sep. Solids - Slurry Trough				7.56	9.58
13.	E O R	Sep. Solids - Solids Trough				8.16	8.98
14.	E O R	Support Frame				0.73	0.72
15.	E O R	Bottom of Lid				6.92	7.53
16.	E O R	Material Crushed Under Scraper				21.67/23.09	25.59/25.89

* BR Before start of Feed to Separator

** E O R End of Run

ANALYSIS OF SAMPLES RUN 2

AMPLE #	TIME Min From Start of Run	TYPE/LOCATION	COMPOSITION				
			OIL	ASPHALTENES	PRE-ASPHALTENES	UNCONV. COAL	ASH
1	7:00	Feed	77.53	14.85	6.98	0.35	0.30/0.50
2	20:00	Separated Liquid	78.10	15.04	6.85	0.01	0.009/0.05
3	22:00	Dispersed Solids	88.58	7.82	1.50	0.97	1.08/1.18
4	23:00	Feed	77.08	14.45	7.35	0.51	0.48/0.54
5	32:00	Separated Liquid	75.35	16.86	7.71	0.08	0.003/0.05
6	33:00	Dispersed Solids	85.32	13.86	-0.85	1.14	0.89/0.18
7	34:00	Feed	75.51	14.91	8.48	0.56	0.52/0.57
8	47:00	Separated Liquid	77.35	14.66	7.95	0.04	0.002/0.06
9	47:00	Dispersed Solids	84.73	8.53	1.33	3.04	2.74/2.00
10	48:00	Feed	77.79	13.05	7.87	0.69	0.59/0.62
11	59:00	Feed	70.48	14.97	11.62	1.89	1.02/1.07
12	58:00	Dispersed Solids	82.79	6.51	4.97	2.03	2.82/3.06
13	58:00	Separated Liquid	76.54	15.07	8.32	0.07	0.00
14	EOR**	Separated Solids - Bowl				26.1	
15	EOR	Separated Solids - Solids Trough	79.67	14.29	5.68	0.18	0.17/0.19
16	EOR	Separated Liquid	87.96	4.42	7.16	0.29	0.13/0.22
17	EOR	Dispersed Solids in vessel	77.12	22.50	-1.24	1.49	0.13
18	EOR	Separated Liquid	82.82	14.55	2.61	0.018	0.00

**EOR End of Run

ANALYSIS OF SAMPLES RUN 3

SAMPLE #	TIME Min From Start of Feed	TYPE/LOCATION	COMPOSITION				
			OIL	ASPHALTENES	PRE-ASPHALTENES	UNCONV. COAL	ASH
–	BR*	FEED				2.91	2.89
1	5:00	Separated Liquid	94.64	4.26	1.04	0.06	0.004/0.0
2	12:00	Feed	87.24	3.76	2.33	3.52	3.17/3.11
3	12:00	Separated Liquid	94.70	4.22	1.08	0.20	0.13/0.04
4	21:00	Feed	86.21	2.65	4.33	3.36	3.29/3.62
5	22:00	Separated Liquid	89.97	5.19	0.84/0.66	2.18/2.36	2.08/2.13
6	25:00	Separated liquid	94.34	3.92	1.22	0.28	0.25/0.23
7	E O R**	Separated Solids - Under Liquid Take Off Ring	39.41	3.28	9.25	21.97	27.17/25.02
8	E O R	Separated Solids - Middle of Bowl Wall	56.22	5.75	7.68	18.15	12.40/11.80
9	E O R	Separated Solids - Bottom of Bowl Wall	44.84	4.42	7.38	16.93	27.24/25.63
10	E O R	Separated Solids - Bottom of Solids Trough	57.86	5.38	7.89	16.82	12.12/11.98
11	E O R	Separated Solids - Top of Solids Trough	53.72	4.17	8.69	16.29	17.18/17.08
12	E O R	Separated Solids - Cross Section of Solids Trough	54.50	4.54	8.66	21.80	15.64/15.32
13	E O R	Separated Solids - Outer Layer of Top of Bowl Wall	56.98	2.64	9.86	18.99	11.34/11.73
14	E O R	Separated Solids - Top of Bowl Wall	53.02	4.29	10.92	21.69	10.10/10.05
15	E O R	Separated Liquid - in Receiving Vessel	92.26	4.09	0.95	0.87	0.82/0.84

*BR Before Start of Feed to Separator

**EOR End of Run

ANALYSIS OF SAMPLES RUN 4

SAMPLE #	TIME Min From Start of Feed	TYPE/LOCATION	COMPOSITION				
			OIL	ASPHALTENES	PRE-ASPHALTENES	UNCONV. COAL	ASH
1	11.0	FEED				4.36	4.12
2	11.0	Separated Liquid				2.88	1.87
3	27.0	Separated Liquid				2.32	1.21
4	34.0	Separated Liquid				3.33	2.96
5	35.0	Feed				4.34	4.39
6	43.0	Separated Liquid				3.70	3.27
7		Separated liquid				2.76	1.56
8	E O R*	Separated Solids - Slurry Trough				2.99	1.22
9	E O R	Separated Solids - Slurry Trough				8.33	12.57
10	E O R	Leakage at Base				3.63	0.88
11	E O R	Separated Solids - Inside Bowl				20.54	36.68
12	E O R	Separated Solids - Slurry Trough				8.67	14.81

*EOR End of Run

ANALYSIS OF SAMPLES RUN 5

AMPLE #	TIME Real	TYPE/LOCATION	COMPOSITION				
			OIL	ASPHALTENES	PRE-ASPHALTENES	UNCONV. COAL	ASH
-	BR*	Vessel 2				9.20	14.41
1	BR*	Feed				2.78	1.85
2	44:00	Separated Liquid				1.99	0.53
3	50:00	Separated Liquid	61.30	21.97	13.85	1.77	1.12/1.09/1.12
4	54:00	Separated Liquid				2.19	0.68
5	60:00	Feed	57.73	24.14	13.96	2.15	2.00/2.08/1.97
6	65:00	Separated Liquid				1.96	0.67
7	69:00	Feed				2.82	1.90
8	69:00	Separated Liquid	61.93	20.67	14.19	1.92	1.27/1.32/1.29
9	EOR**	Separated Liquid in vessel				2.23	1.01
10	EOR**	Separated Liquid in vessel	60.17	22.16	14.67	1.83	1.13/1.15/1.22
11	EOR	Feed	57.21	16.35	10.26	6.82	9.38/9.35/9.35
12	EOR	Separated Solids - Liquid Take-off Ring				19.95	30.06

*BR Before start of Feed to Separator

**EOR End of Run

ANALYSIS OF SAMPLES RUN 6

SAMPLE #	TIME Min From Start of Feed	TYPE/LOCATION	COMPOSITION OIL	ASPHALTENES	PRE-ASPHALTENES	UNCONV. COAL	ASH
-	BR *	Feed	60.86			2.80	3.25/3.24
1	2:00	Feed				3.15	3.28
2	7:00	Separated Liquid				1.54	0.70
3	11:00	Feed				3.12	3.23
4	15:00	Separated Liquid				1.88	1.62
5	19:00	Separated Liquid				1.92	1.32
6	21:00	Separated Liquid					
7	26:00	Separated Liquid Feed				1.41	0.64
8	28:00	Feed				3.09	3.29
9	31:00	Separated Liquid				1.10	0.49
10	35:00	Separated Liquid				2.11	1.96
11	35:00	Feed				3.06	3.36
12	EOR**	Dispersed Solids - in vessel				4.52	6.97
13	EOR	Separated Liquid - in vessel				1.74	1.59
14	EOR	Under Slurry Trough				1.69	0.63

* BR Before Start of Feed to Separator

** EOR End of Run

ANALYSIS OF SAMPLES RUN 7

SAMPLE #	TIME Min From Start of Run	TYPE/LOCATION	COMPOSITION				
			OIL	ASPHALTENES	PRE-ASPHALTENES	UNCONV. COAL	ASH
1	BR*	FEED	64.98	18.44	11.62	2.46	2.50
2	3:00	Separated Liquid	69.66	16.86	11.89	1.19	0.40
3	6:00	Separated Liquid	66.24	17.44	15.15	1.17	0.17
4	8:00	Separated Liquid	70.99	16.35	11.33	1.17	0.16
5	12:00	Separated Liquid	66.20	17.99	13.94	1.87	0.28
6	15:00	Separated Liquid	65.24	20.54	12.95	1.27	0.14
7	18:00	Feed	60.56	20.91	12.97	2.96	2.60
8	19:00	Separated Liquid	62.14	20.02	9.60	4.62	3.62
9	25:00	Separated Liquid	66.85	13.62	11.21	1.79	0.95
10	30:00	Feed	61.63	21.57	11.19	2.91	2.60
11	30:00	Separated Liquid in vessel	64.78	20.73	11.08	3.41	1.25
-	EOR**	Separated Solids - Lower Bowl Wall	33.59	6.86	15.00	19.94	24.61
-	EOR	Separated Solids - Ridge under pusher	33.24	6.85	20.33	12.90	27.68
-	EOR	Separated Solids - Slurry Trough	40.39	10.46	14.80	15.65	18.70
-	EOR	Feed - Bottom of - vessel	61.23	22.86	9.20	3.73	2.98

*BR Before Start of Feed to Separator

** EOR End of Run

ANALYSIS OF SAMPLES RUN 8

MPLE #	TIME Min From Start of Feed	TYPE/LOCATION	COMPOSITION				
			OIL	ASPHALTENES	PRE-ASPHALTENES	UNCONV. COAL	ASH
1	2:00	Separated Liquid	69.24	20.20	10.05	0.43	0.02/0.04/0.18
2	9:00	Separated Liquid	67.33	21.05	10.56	0.73	0.25/0.27/0.47
3	12.40	Separated Liquid	71.20	14.95	9.87	8.07	1.78/1.75/1.84
4	17:00	Separated Liquid	67.86	19.77	10.91	0.99	0.50/0.49/0.42
5	19:30	Separated Liquid	NA	NA	11.71	0.65	0.07/0.31/0.32
6	24:00	Separated Liquid	70.13	17.32	10.99	0.87	0.63/0.75/0.70
7	26:00	Separated Liquid	71.29	16.87	10.48	0.79	0.49/0.67/0.54
8	28:00	Feed in vessel	63.03	21.40	8.86	3.08	3.52/3.72/3.65
9	28:00	Separated Liquid	67.86	21.00	10.13	0.79	0.16/0.25/0.26
10	EOR **	Separated Liquid - in vessel	67.18	19.01	10.94	1.68	1.18/1.18/1.21
-	EOR	Separated Solids - Top of Bowl	21.04	7.02	6.70	46.37	43.72/NA/49.02
-	EOR	Separated Solids - Middle of Bowl	38.72	16.31	9.64	17.02	18.21/18.72/17.99
-	EOR	Separated Solids - Bottom of Bowl	30.32	11.70	1.63	17.27	40.08/39.25/37.91
-	EOR	Separated Solids - Under Lid	39.18	12.00	11.16	14.58	21.84/22.19/25.21
-	EOR	Separated Solids - Under Vessel	68.08	19.53	10.30	0.87	0.89/0.83/1.94
-	EOR	Separated Solids - Under Vessel	70.08	18.74	9.24	0.88	0.80/1.16/1.23

** EOR End of Run

ANALYSIS OF SAMPLES RUN 9

AMPLE #	TIME Min From Start of Feed	TYPE/LOCATION	COMPOSITION				
			OIL	ASPHALTENES	PRE-ASPHALTENES	UNCONV. COAL	ASH
1	1:40	Separated Liquid				0.39	0.10
2	2:30	Separated Liquid				0.51	0.02
3	5:00	Separated Liquid				0.54	0.29
4	8:20	Separated Liquid				0.59	0.12
5	9:40	Separated Liquid				0.67	0.09
6	13:00	Separated Liquid				1.34	0.64
7	15:00	Separated Liquid				4.15	1.01
8	18:20	Separated Liquid				0.51	0.13
9	20:20	Separated Liquid				1.65	1.66
10	24:16	Separated Liquid				0.96	0.19
11	28:49	Separated Liquid				3.15	3.03
12	32:03	Separated Liquid				2.74	2.46
13	25:48	Separated Liquid				2.86	2.65
14	35:04	Separated Liquid				1.96	1.37
15	38:40	Feed				2.99	3.78
16	40:40	Separated Liquid				1.72	0.50
17	41:40	Separated Liquid				9.71	9.46
18	43:20	Separated Liquid				1.59	0.40
19	46:00	Separated Liquid				1.42	0.10
20	47:40	Separated Liquid				1.40	0.24
21	EOR**	Separated Liquid in Vessel				2.03	0.95

** EOR End of Run

ANALYSIS OF SAMPLES RUN 9 (Cont'd)

SAMPLE #	TIME From Start of Feed	TYPE/LOCATION	COMPOSITION				
			OIL	ASPHALTENES	PRE-ASPHALTENES	UNCONV. COAL	ASH
-	EOR**	Separated Solids - Bowl Top				22.74	39.95
-	EOR	Separated Solids - Bowl Middle				15.87	21.21
-	EOR	Separated Solids - Top of Scraper				1.23	0.82
-	EOR	Slurry Trough Solids				17.15	27.31
-	EOR	Slurry Trough Liquid				1.96	1.92
-	EOR	Housing Channel Drain "A"				1.04	0.43
-	EOR	Housing Channel Drain "B"				0.83	0.03

**EOR End of Run

ANALYSIS OF SAMPLES RUN 10

SAMPLE #	TIME Min From Start of Feed	TYPE/LOCATION	COMPOSITION				
			OIL	ASPHALTENES	PRE-ASPHALTENES	UNCONV. COAL	ASH
1	0:25	Separated Liquid				1.17	0.30
2	0:40	Separated Liquid				0.70	0.45
3	2:43	Separated Liquid				1.14	0.16
4	6:20	Separated Liquid				1.40	0.83
5	6:50	Separated Liquid				1.31	0.22
6	7:47	Separated Liquid				0.84	0.008
7	9:45	Separated Liquid				1.16	0.82
8	10:25	Separated Liquid				0.98	0.48
9	11:20	Separated Liquid				0.71	0.18
10	13:15	Separated Liquid				1.51/2.01	0.99/1.26
11	14:00	Separated Liquid				1.08/0.54	0.83/1.19
12	15:15	Separated Liquid				0.76	0.12
13	18:35	Separated Liquid				1.18/1.52	0.60/0.33
14	20:15	Separated Liquid				0.86/1.09	0.33/0.42
15	21:30	Separated Liquid				2.52	2.03
16	25:00	Separated Liquid				1.25	0.30
17	27:15	Separated Liquid				1.28	0.70
18	EOR **	Feed				1.76	1.28
19	EOR	Separated Liquid in Vessel				1.34	0.49

** EOR End of Run

ANALYSIS OF SAMPLES RUN 10 (Cont'd)

SAMPLE #	TIME	TYPE/LOCATION	COMPOSITION				
			OIL	ASPHALTENES	PRE-ASPHALTENES	UNCONV. COAL	ASH
-	BR*	Feed					2.83
-	EOR**	Separated Solids - Top of Bowl				26.38	18.22
-	EOR	Separated Solids - Middle Of Bowl				16.17	15.82
-	EOR	Separated Solids - Bottom of Bowl				22.31	21.46
-	EOR	Separated Solids - Inner Wall of Bowl				20.79	20.72
-	EOR	Separated Solids - Under Lid				20.13	17.86
-	EOR	Separated Solids - Slurry Trough Solids				13.46	13.50
-	EOR	Separated Solids - Top of Scraper				3.29	2.52
-	EOR	Separated Solids - Slurry Trough				13.71	13.46
-	EOR	Separated Solids - Slurry Trough Liquid				1.98	0.11
-	EOR	Separated Solids - Bottom of Ext. Tank				0.91	0.32
-	EOR	Separated Solids - Housing Drainage Channel				0.84	0.21

*BR Before Start of Feed to Separator

*EOR End of Run

Appendix 4

Equipment Details

Instrument List

Control Panel Layout

Control Panel Layout

PLC Programming Details

CONFIDENTIAL

RUN 7 LAB SEPARATION #1

CONDITIONS - Starting material vessel 1 Run 7
- Centrifuge at 180°C 5 minute, max rpm immediately after sampling

RESULTS

	MASS g.	COMPOSITION				
		Oil	Asphaltene	Pre Asphaltene	Unconverted Coal	Ash
Starting Material	NA					
Top	21.8504	65.88	21.93	11.02	1.02	0.02/0.04/0.09
Bottom	3.6281	40.54	11.09	11.42	17.14	20.43/20.31/18.68

COMMENTS

Bottoms - flat, dull, grey, black at room temperature.

RUN 7 LAB SEPARATION #2

CONDITIONS - Starting material vessel 1 Run 7
- Centrifuge at 150°C 5 minute, max rpm. immediately after sampling

RESULTS

	MASS g.	COMPOSITION				
		Oil	Asphaltene	Pre Asphaltene	Unconverted Oil	Ash
Starting Material	NA					
Top	25.1945	68.31	19.22	11.62	0.79	0.04/0.05/0.09
Bottom	5.4713	44.23	11.36	15.34	13.64	15.40/17.06/13.84

COMMENTS

Bottoms - flat, dull, grey, black at room temperature.

RUN 7 LAB SEPARATION #3

CONDITIONS - Starting material vessel 1 Run 7
- Separate at 180°C 5 minutes, max rpm
(after sampling allow to cool down then
plunge into 180°C oil bath, stir for 10 min).

RESULTS

	MASS g.	COMPOSITION				
		Oil	Asphaltene	Pre Asphaltene	Unconverted Coal	Ash
Starting Material	33.5395					
Top	28.3548	62.63	23.99	12.37	0.97	0.04/0.08/NA
Bottom	4.6275	40.06	15.23	9.21	16.14	19.61/19.36/19.55
Lost	0.5572					

COMMENTS

Separated Solids - Flat dull grey black at room temperature.

RUN 8 LAB SEPARATION

CONDITIONS
- Starting material vessel 1 Run 8
- centrifuge 180°C For 5 minute, max rpm
- material processed immediately after sampling

RESULTS

	MASS g.	COMPOSITION Oil	Asphaltene	Pre Asphaltene	Unconverted Coal	Ash
Starting Material	25.7733					
Top	20.3764	87.18	3.91	8.65	0.26	0.004/N/A/-
Bottom	5.3969	32.44	10.23	19.03	15.53	21.70/23.21/23.39
Lost	-					

COMMENTS

Separated Solid - flat, dull, grey, black at room temperature.

RUN 9 LAB SEPARATION

CONDITIONS
- Starting material vessel 1 Run 9
- separate at 180°C For 5 minute, max rmp
- material processed immediately after sampling

RESULTS

	MASS g.	COMPOSITION				
		Oil	Asphaltene	Pre Asphaltene	Unconverted Coal	Ash
Starting Material	22.8996					
Top	18.9245	77.82	13.35	8.20	0.57	0.08/0.02/0.07
Bottom	3.9751	46.79	5.61	8.00	18.38	22.06/21.31/20.29

COMMENTS

Separated Solid - flat, dull, grey, black at room temperature.

Appendix 5

Analytical Procedures

CONFIDENTIAL

Equipment Details

Feed and Product Vessels

316 - Stainless Steel Construction
- Body 3/16" thick
- top 1/4" thick
- lid 1/4" thick
- nominal volume 112L - useable volume 104.6111 L.
- bolt and gasket lid
- dish bottom
- 32" x 16" round
- 40 of 5/8 tube coiled around body and incased in 7/8" of heat conductive cement
- 3000 W - 240 V heating jacket with insulated bottom
- "J" type thermocouples installed in cement and inside vessel.
- Capacitive type teflon level probes with top mounted transmitter
- 5/8 tubing to 3/8" stainless steel educator mounted close to bottom of dish
- Gravity fed rotor gear pump mounted under vessel with carbon bushings and grafoil packaging for high temperature use drivers by 1/2 H.P. D.C. permanent magnetic motor through 3:1 gear reducer and flexible coupling

Feed Buffer Tank

- 316 stainless steel construction
- Body 1/8" thick
- Top 3/16" thick
- Cover 3/16" thick
- Gasketed bolted lid
- Conical shape
- 12" high x 12" deep
- 12" capacitive type teflon level probe with transmitter mounted on top

Valves

Manual - 1/2" N.P.T. 304 and 316 stainless steel
- teflon seals

Remote

- electric continuous turn 1/2 stainless steel ball valve 115V motor actuator.

- 1 electric 90° 3/8" stainless steel ball valve with 4-20 MA electronic positioner.

- 1 air to open 1/2" N.P.T. vee steel stainless steel control valve with I to P converter.

Tubing, Piping and Fittings

- All tubing and piping 316 stainless steel in sizes 3/4" NPT, 1/2" NPT, 1/4" tube, 3/8" tube, 1/2 tube and 5/8" tube.

- All tube fittings 316 stainless steel nut and ferrule type.

- All pipe fittings cast 304 stainless steel 150 lb.

Instrument List

Field

Flow

1 - 316 stainless steel turbine flow meter with pulse output transmitter.

1 - 316 stainless steel Stuaff gear type flow meter with pulse output transmitter.

Temperature

18 - "J" type thermocouples

Level

4 - Vessels 1, 2, 3, 4 Robertshaw frequencey - capacitance type level probes,
1 - Buffer tank

Panel Temperature

18 temperature controllers "J" type thermocouples. Range 0-400°C. Relay output to control. Breakdown:

4-loops controlling the temperature of the vessels V1 to V4 composed of two controllers with their outputs interlocked. Controller one indicated the temperature of the inside liquid and controller two indicated the outside temperature of the vessel under the heating jacket.

5-loops controlling the heat tracing on various tubing and piping. The thermocouples were placed under the heat tracing between the heat tracing and process tubing.

1-loop controlling the heat tracing on the pump housing with the thermocouple under the tracing and between the tracing and the body of V1 pump.

1-loop controlling the temperature of the centrax with two controllers with their outputs interlocked. Controller 1 indicated the temperature inside the centrax. Controller 2 indicated the temperature of the body under the heating tape.

1-loop controlling the temperature inside the drain trough under the separator bowl. The thermocouple was part of the heater assembly.

2-loops indicating the temperature of the centrax bearings. The thermocouples were positioned on the house directed on top of the bearings.

Panel (Cont'd)

Variable Speed

6-S.L.R. type D.C. speed controllers controlling mixer, transfer, and metering pump speeds.

1-S.C.R. type 7.5 H.P. D.C. speed control with D.C. ammeter and tachometer controlling the centrax speed.

Programmable Micro Processors

1-G.E. series one P.L.C. with one 24VDC output cards and two input cards controlled the centrax pusher interfaced with a dixon three trip positioner, a micro-flow flow processor and a Stauff gear type flow meter.

1-Micro-flow flow processor which received pulses from the Stauff flow meter and two turbine flow meters, performed the necessary mathematics with given parameters inputed through the keyboard. 4-20 MA control signals were outputed to two control valves and the positioner.

4-Microprocessor based level indicators received signals from probes and transmitters mounted on V1, V2, V3 and V4.

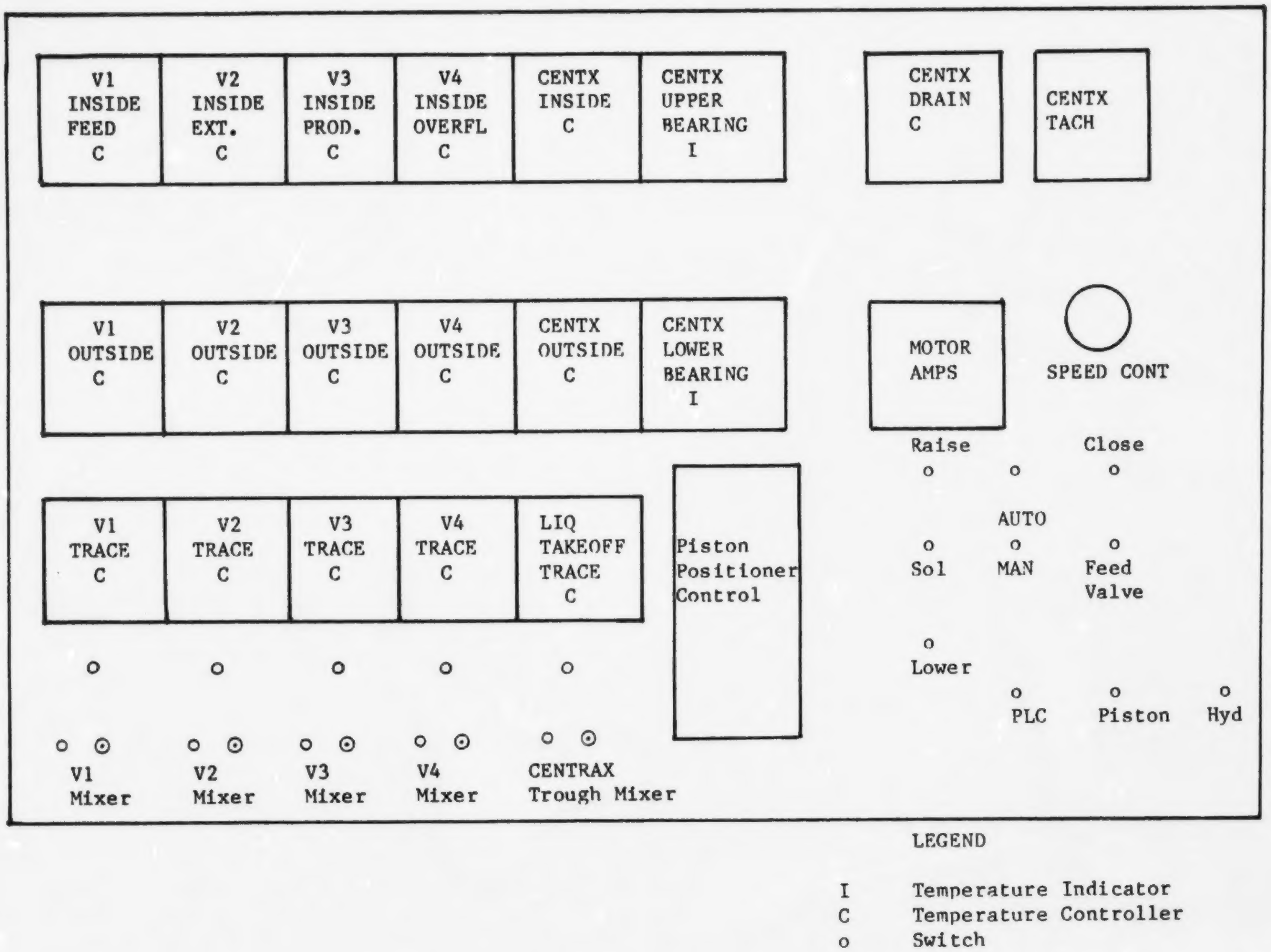
1-Microprocessor based level alarm received signals from a level probe and transmitter mounted in the buffer vessel V5.

Miscellaneous

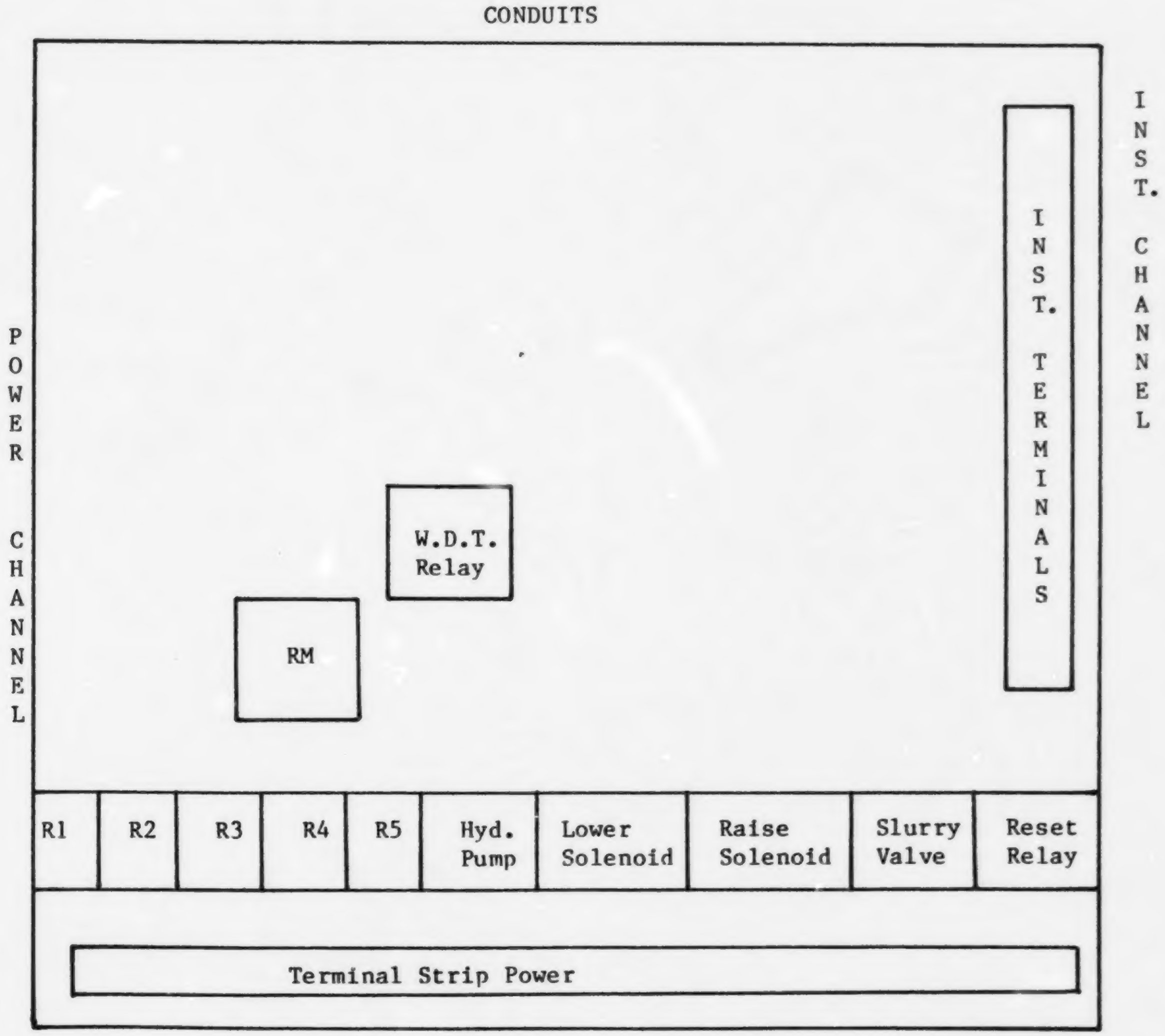
6 - 120V control relays 2 pole
6 - 24V control relays 2 pole
1 - 120V control relays 4 pole
2 - electromagnetic totalizers.

Control Panel Layout

LAYOUT OF CONTROL PANEL


V1 INSIDE FEED C
V2 INSIDE EXT. C
V3 INSIDE PROD. C
V4 INSIDE OVERFL C
CENTX INSIDE C
CENTX UPPER BEARING I
CENTX DRAIN C
CENTX TACH
V1 OUTSIDE C
V2 OUTSIDE C
V3 OUTSIDE C
V4 OUTSIDE C
CENTX OUTSIDE C
CENTX LOWER BEARING I
MOTOR AMPS
SPEED CONT
V1 TRACE C
V2 TRACE C
V3 TRACE C
V4 TRACE C
LIQ TAKEOFF TRACE C
Piston Positioner Control
Raise
Close
AUTO
Sol
MAN
Feed Valve
Lower
PLC
Piston
Hyd
V1 Mixer
V2 Mixer
V3 Mixer
V4 Mixer
CENTRAX Trough Mixer
LEGEND
I Temperature Indicator
C Temperature Controller
o Switch

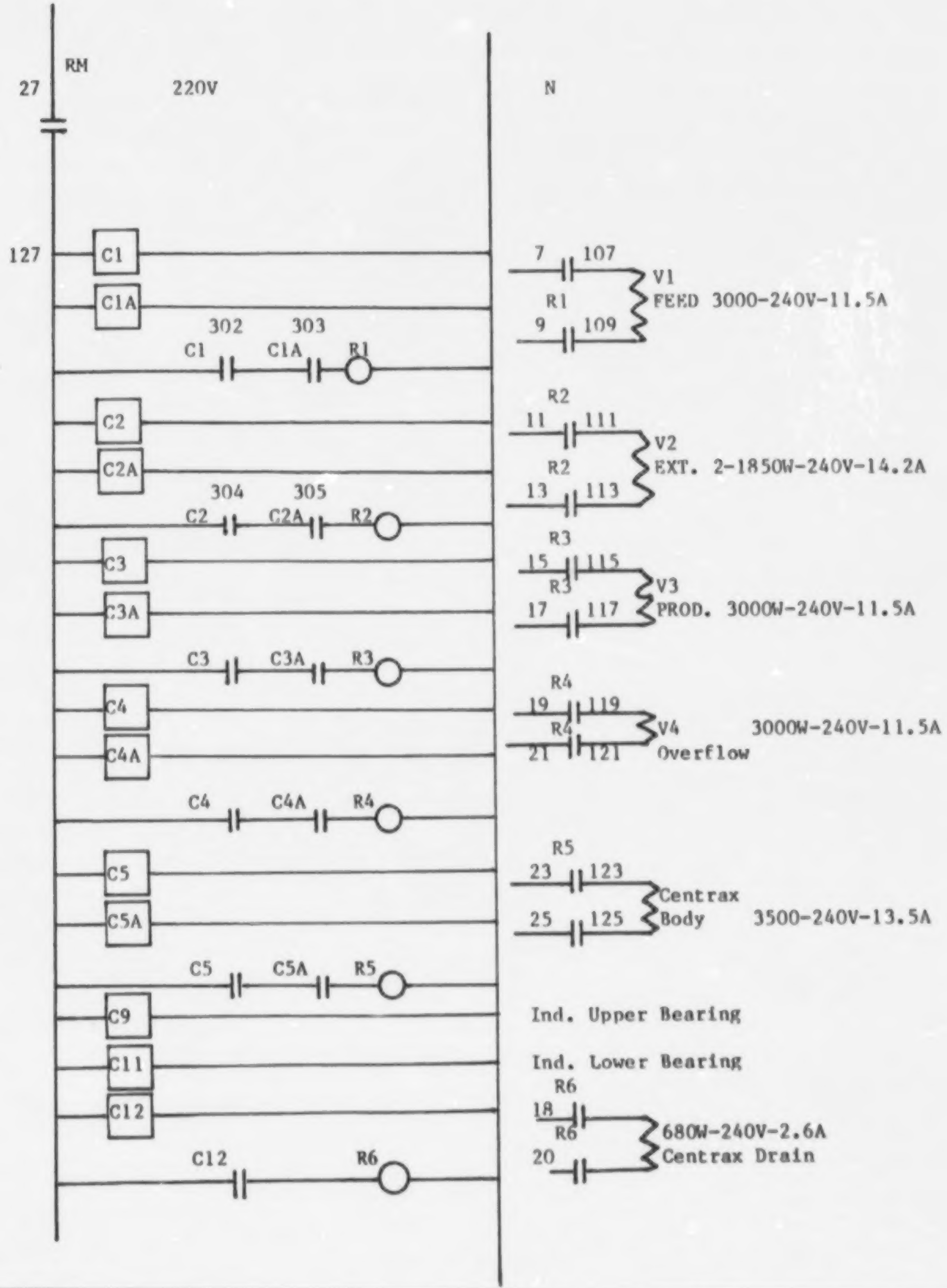
TERMINAL & RELAY LAY-OUT OF CONTROL PANEL


CONDUITS
POWER CHANNEL
INST. CHANNEL
INST. TERMINALS
W.D.T. Relay
RM
R1
R2
R3
R4
R5
Hyd. Pump
Lower Solenoid
Raise Solenoid
Slurry Valve
Reset Relay
Terminal Strip Power

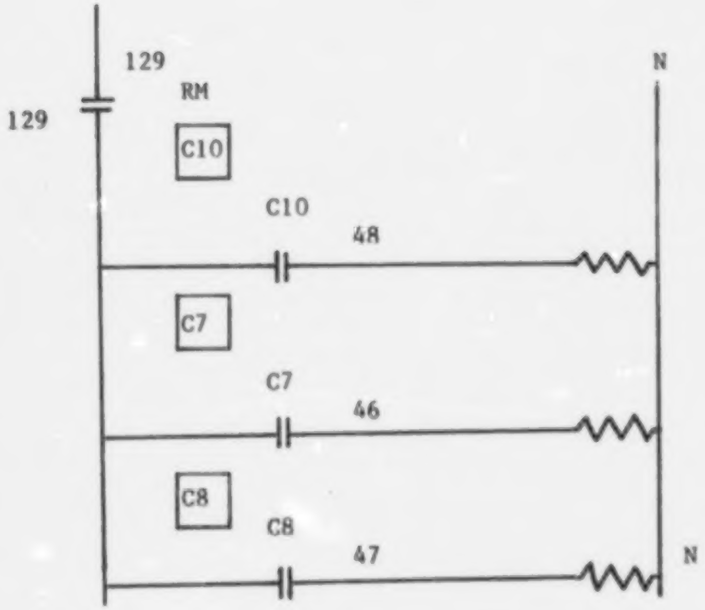
DISTRIBUTION OF CONTROL PANEL CIRCUITS

	Voltage	From	To	Wire & Amp	Colour
Feed tank heating	220	P.P.B.	Centrax Cont.	7 - 20	Red
	220	"	"	9 - 20	Black
Ext. tank heating	220	"	"	11 - 20	Blue
	220	"	"	13 - 20	Red
Product tank heating	220	"	"	15 - 20	Black
	220	"	"	17 - 20	Blue
Slurry tank heating	220	"	"	19 - 20	Red
	220	"	"	21 - 20	Black
Separation Body	220	"	"	23 - 20	Blue
	220	"	"	25 - 20	Red
Panel Power Cir. #1	120	"	"	27 - 20	Black
Panel Power Cir. #2	120	"	"	29 - 20	Blue
Panel Power Cir. #3	120	"	"	8 - 20	Red
Motor Controls Cir #5	220	"	"	10 - 20	Black
	220	"	"	12 - 20	Blue
Motor Controls Cir #6	220	"	"	14 - 20	Red
	220	"	"	16 - 20	Black
Motor Controls Cir #7	220	"	"	18 - 20	Blue
	220	"	"	20 - 20	Red
Panel Power Cir #4	120	"	"	22 - 20	Black
Hyd. Solenoids & Slurry Valve	120	"	"	24 - 20	Blue
Motor Controls 7.5 H.P. Centrax	220	"	"	26 - 40	Red
	220	"	"	28 - 40	Black
	220	"	"	30 - 40	Blue

CONTROL PANEL SCHEMATIC


27
RM
220V
N
127
C1
C1A
302
303
C1
C1A
R1
7
107
V1
R1
FEED 3000-240V-11.5A
9
109
C2
C2A
304
305
C2
C2A
R2
R2
11
111
V2
R2
EXT. 2-1850W-240V-14.2A
13
113
C3
C3A
C3
C3A
R3
R3
15
115
R3
V3
17
117
PROD. 3000W-240V-11.5A
C4
C4A
C4
C4A
R4
R4
19
119
R4
V4
3000W-240V-11.5A
21
121
Overflow
C5
C5A
C5
C5A
R5
R5
23
123
Centrax
25
125
Body
3500-240V-13.5A
C9
Ind. Upper Bearing
C11
Ind. Lower Bearing
C12
C12
R6
R6
18
R6
680W-240V-2.6A
20
Centrax Drain

CONTROL PANEL SCHEMATIC (Cont'd)

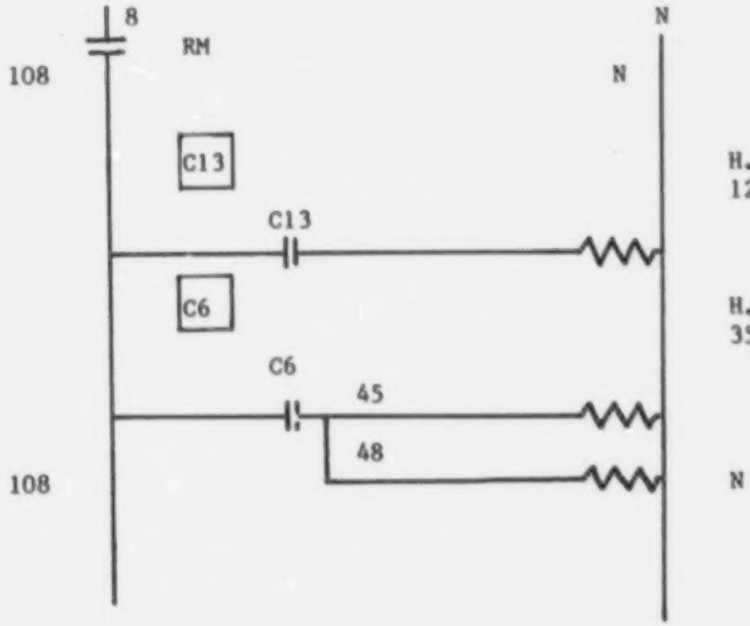

129
RM
129
C10
C10
48
C7
C7
46
C8
C8
47
N
N

Circuit 2

Product H.T. V3
350W 350W - 120V - 5.8 A

H.T. Feed lines V1 Meter Pump
580W - 120V - 4.8 A

H.T. Ext. Lines V2
350W - 120V - 2.9 A

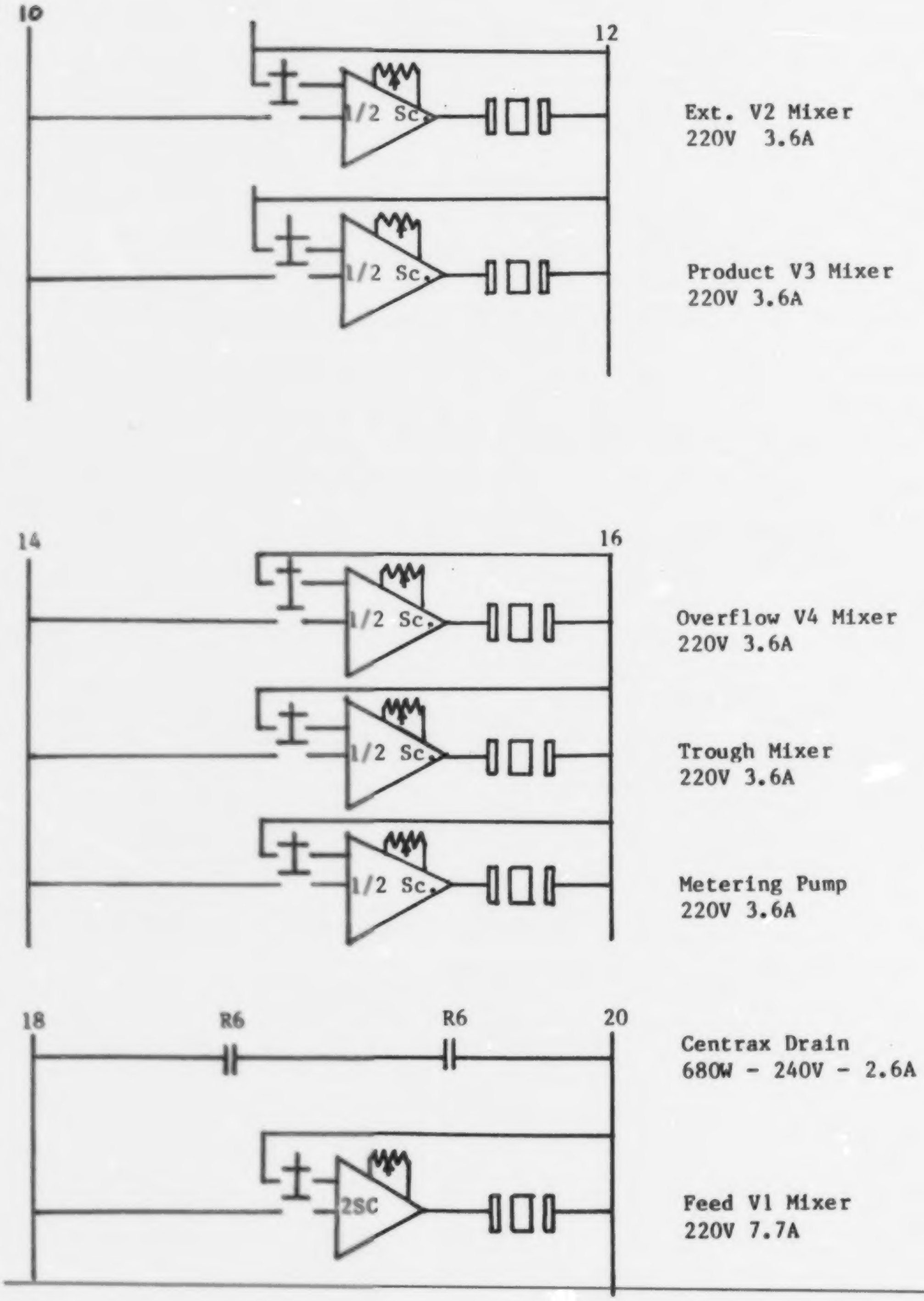

8
RM
108
N
N
C13
C13
C6
C6
45
48
108
N

Circuit 3

H.T. Liquid Takeoff
120V-300W - 2.5 A

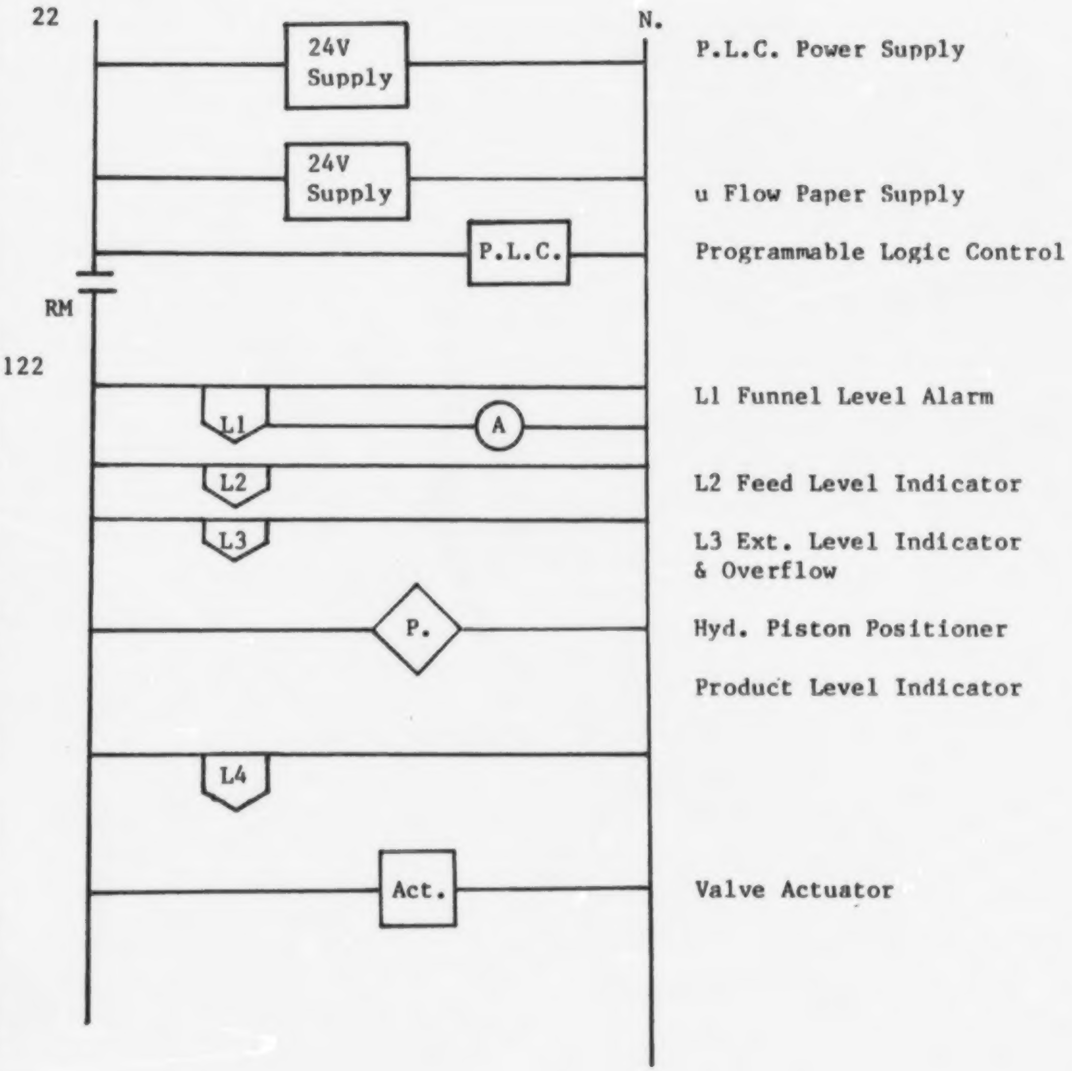
H.T. Overflow V4 Pumps
350W 350W 580W - 120V-10.7A

CONTROL PANEL SCHEMATIC (Cont'd)


10
12
1/2 Sc.
Ext. V2 Mixer
220V 3.6A
1/2 Sc.
Product V3 Mixer
220V 3.6A
14
16
1/2 Sc.
Overflow V4 Mixer
220V 3.6A
1/2 Sc.
Trough Mixer
220V 3.6A
1/2 Sc.
Metering Pump
220V 3.6A
18
R6
R6
20
Centrax Drain
680W - 240V - 2.6A
2SC
Feed V1 Mixer
220V 7.7A

CONTROL PANEL SCHEMATIC (Cont'd)

Circuit # 4


22
N.
24V Supply
P.L.C. Power Supply
24V Supply
u Flow Paper Supply
P.L.C.
Programmable Logic Control
RM
122
L1
A
L1 Funnel Level Alarm
L2
L2 Feed Level Indicator
L3
L3 Ext. Level Indicator & Overflow
P.
Hyd. Piston Positioner
Product Level Indicator
L4
Act.
Valve Actuator

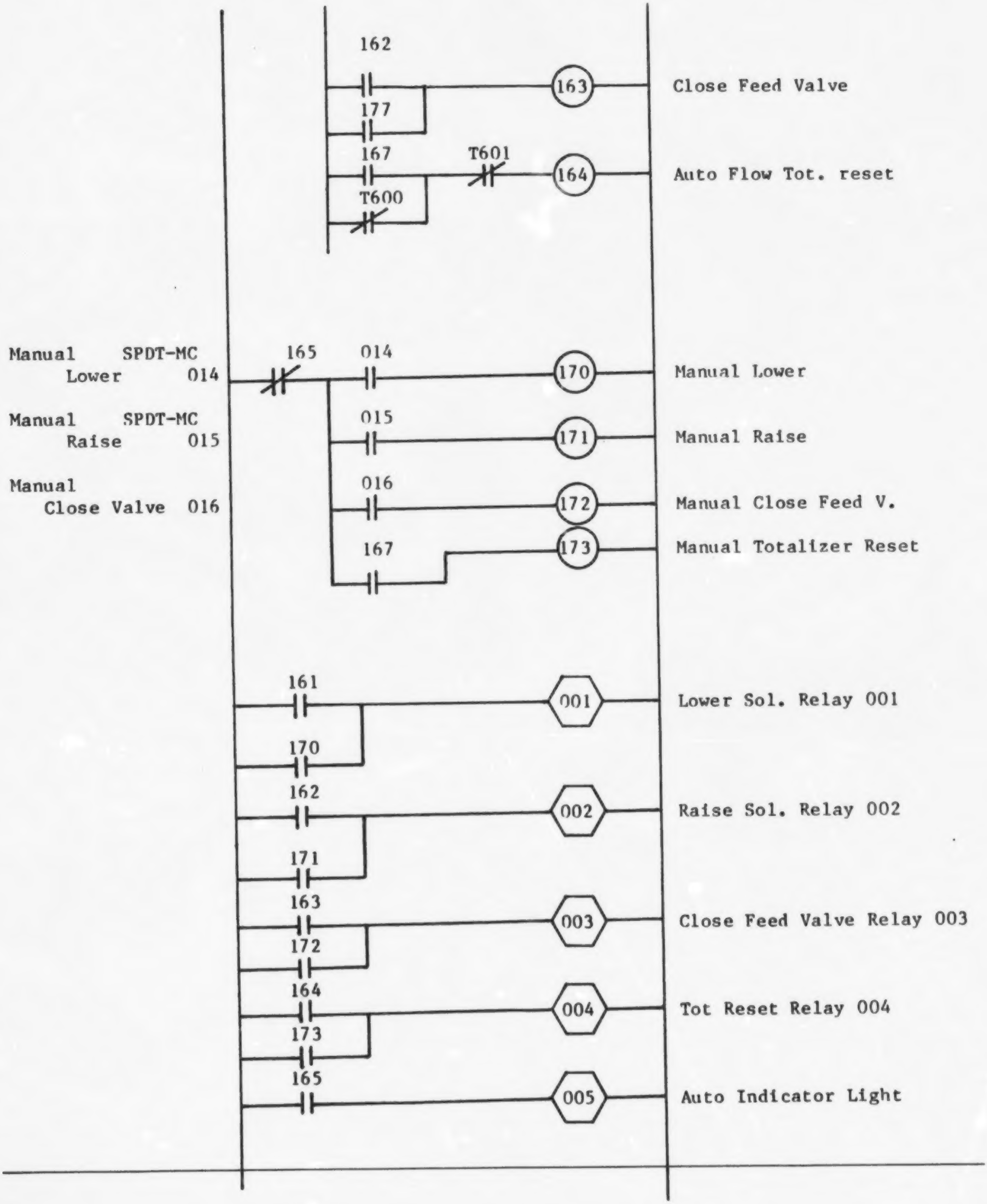
P.L.C. Programming Details

LIST PLC I/O ALLOCATION

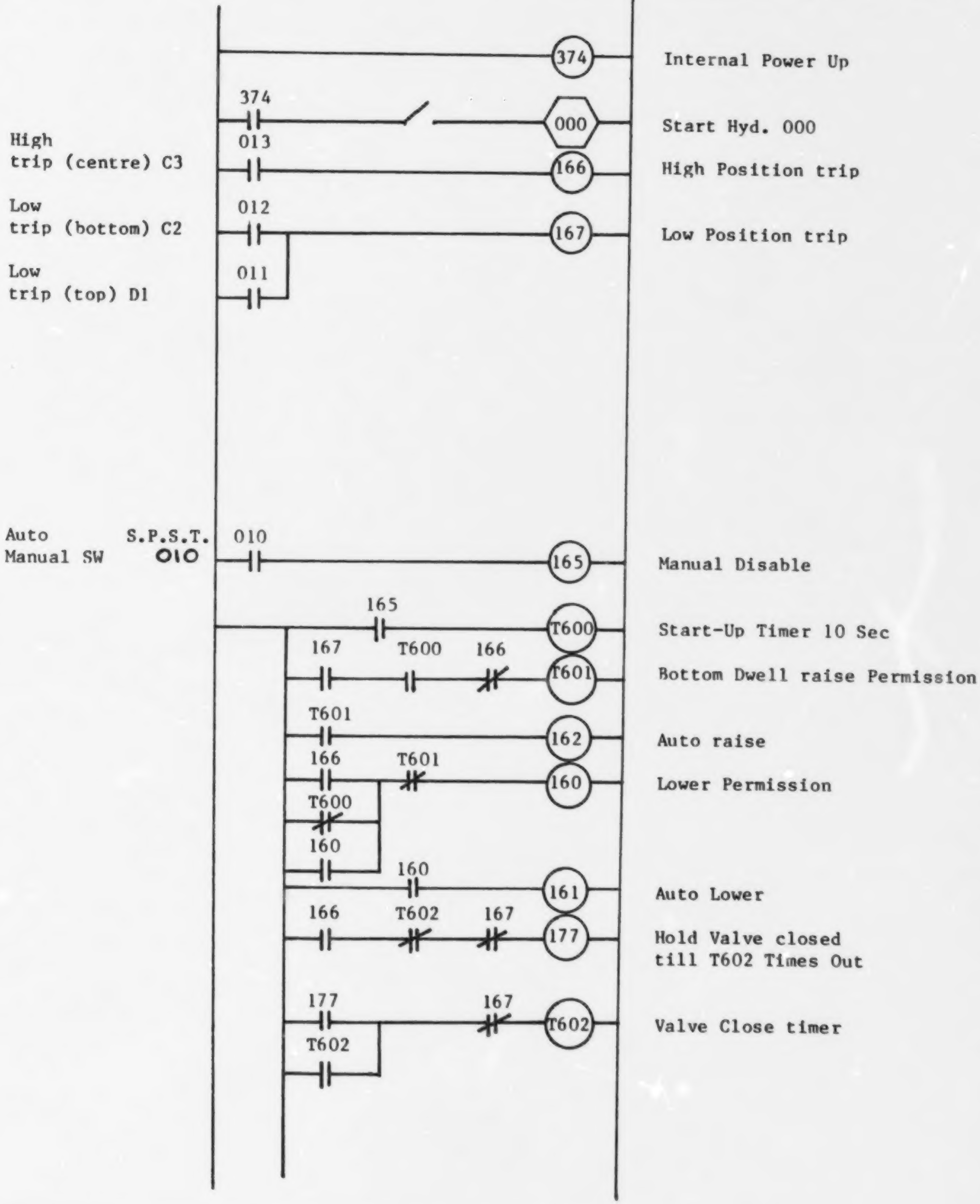
Function	#	Description
Output	000	Start hyd. pump to 24V relay to hyd. starter
Output	001	Lower sol. valve from 24V relay
Output	002	Raise sol. valve from 24V relay
Output	003	Close feed valve 24V relay with open close contact bridged
Output	004	Totalize reset to 24V relay
Output	005	Auto Light
Output	006	Miro Flow Power Relay (W.D.T.)
Output	007	
Input	010	Auto/Manual on-off switch
Input	011	Low trip 20MA set point from controller (return to bottom)
Input	012	Low trip 4MA set point from controller (real bottom)
Input	013	High trip mid-point on controller
Input	014	Manual lower - SPDT-M.C. also manual raise
Input	015	Manual raise other pole of manual lower switch
Input	016	Manual on-off toggle
Input	017	W.D.T.
Input	020	
Input	021	
Input	022	
Input	023	
Input	024	
Input	025	
Input	026	
Input	027	

```
STR
374  E
OUT
000  E
STR
013  E
OUT
166  E
STR
012  E
OR
011
OUT
167  E
STR
010  E
MCS  E
OUT
165  E
TMR
600  E
- 10  E
STR
167  E
OR
TMR
601  E
AND
NOT
166  E
TMR
601  E
- 95  E
STR
TMR
601  E
OUT
162  E
STR
166  E
OR
NOT
TMR
600  E
OR
160  E
AND
NOT
TMR
601  E
OUT
160  E
STR
160  E
OUT
161  E
STR
166  E
AND
NOT
167  E
AND
NOT
TMR
602  E
OUT
171  E
STR
177  E
OR
TMR
602  E
AND
NOT
167  E
TMR
602  E
-  2  E
STR
162  E
OR
177  E
OUT
163  E
STR
167  E
OR
NOT
TMR
600  E
AND
NOT
TMR
601  E
OUT
164  E
MCR  E
STR
NOT
165  E
MCS  E
STR
014  E
OUT
170  E
STR
015  E
OUT
STR
016  E
OUT
172
STR
167  E
OUT
173  E
MCR  E
STR
161  E
OR
170  E
OUT
001
STR
162  E
OR
171  E
OUT
002  E
STR
163  E
OR
172  E
OUT
003  E
STR
164  E
OR
173  E
OUT
004  E
STR
165  E
OUT
005  E
STR
017  E
OUT
200  E
STR
200  E
TMR
603  E
-  1  E
STR
200  E
AND
NOT
TMR
603  E
OUT
201  E
STR
NOT
201  E
OUT
006  E
```

PLC LADDER DIAGRAM

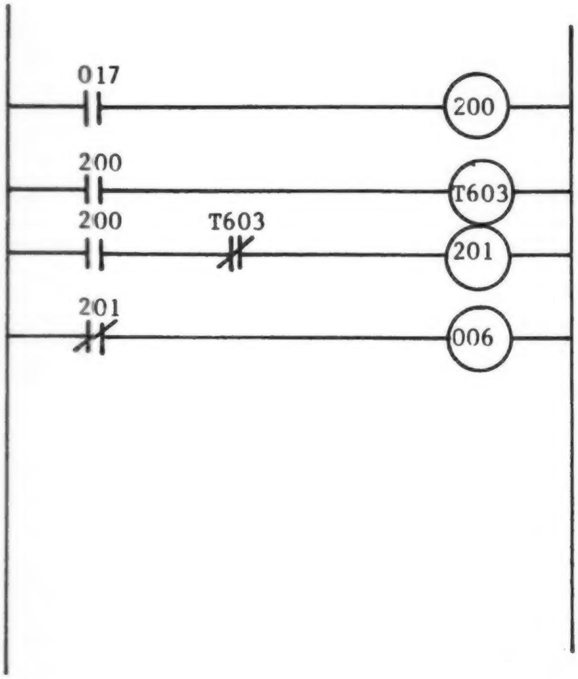

162
163
Close Feed Valve
177
167
T601
164
Auto Flow Tot. reset
T600
Manual SPDT-MC
Lower 014
Manual SPDT-MC
Raise 015
Manual
Close Valve 016
165
014
170
Manual Lower
015
171
Manual Raise
016
172
Manual Close Feed V.
167
173
Manual Totalizer Reset
161
001
Lower Sol. Relay 001
170
162
002
Raise Sol. Relay 002
171
163
003
Close Feed Valve Relay 003
172
164
004
Tot Reset Relay 004
173
165
005
Auto Indicator Light

PLC LADDER DIAGRAM (Cont'd)


374
Internal Power Up
374
000
Start Hyd. 000
High
trip (centre) C3
013
166
High Position trip
Low
trip (bottom) C2
012
167
Low Position trip
Low
trip (top) D1
011
Auto
Manual SW
S.P.S.T.
010
010
165
Manual Disable
165
T600
Start-Up Timer 10 Sec
167
T600
166
T601
Bottom Dwell raise Permission
T601
162
Auto raise
166
T601
160
Lower Permission
T600
160
160
161
Auto Lower
166
T602
167
177
Hold Valve closed
till T602 Times Out
177
167
T602
T602
Valve Close timer

PLC LADDER DIAGRAM (Cont'd)

W.D.T. 017


017
200
200
T603
200
T603
201
201
006

Timer 1 sec for reset

U Flow Power 006

Appendix 6

Typical Output for Economic Model

Case B-1

CONFIDENTIAL

ANALYTICAL PROCEDURES

Sampling Techniques

Generally, the materials handled such as: feed slurry, heavy product, light product and residue are heterogeneous. In order to obtain representative samples for analysis these materials must be thoroughly homogenized.

The sampling method employed is dependent on the nature of the sample at room temperature. Feed and heavy product were either heated to a liquid phase or cooled in the feezer and crushed with a mortar and pestle prior to mixing and sampling.

The light products are generally composed of two phases. These are separated into an aqueous fraction and an oil fraction and samples are taken of each for analysis.

METHODS OF ANALYSIS

Solvent Extraction

The products are considered to consist of solids, pre-asphaltenes, asphaltenes and oil. These are generally characterized by their solubility in different solvents.

In this report, solids are considered to be the fraction which is insoluble in tetrahydrofuran (THF) and consists of coke like material and catalyst; pre-asphaltenes are those that are soluble in THF but insoluble in toluene; asphaltenes are soluble in toluene but insoluble in n-heptane. The heptane soluble portion is considered to be maltene.

The composition of each sample is determined by sequential extraction with solvents and then determining each fraction by difference as outlined earlier. Solvent extractions and insoluble collection is performed by vacuum filtration which allows for immediate analysis. Individual samples are used for each analysis.

For each analysis a sample is weighed in a beaker (approx. 1g) and dissolved by an excess (approx. 50 times the sample weight) of the appropriate solvent.

Maximum solvent extraction is accomplished by stirring the sample vigorously on a hot plate near the boiling point of the solvent for a minimum of thirty minutes.

The sample is allowed to cool to room temperature and then is filtered under vacuum using a preweighed glass fiber pad (whatman glass microfilter). The filter cake is washed with an excess amount of solvent and then dried in an oven until instant weight is achieved.

VACUUM DISTILLATION

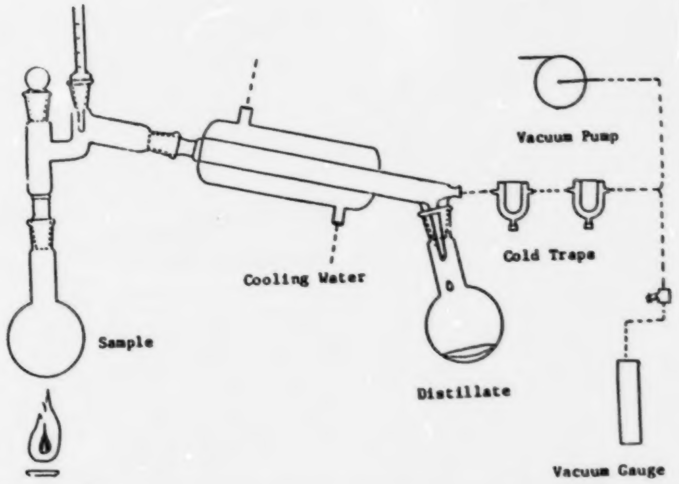
The apparatus used to carry out the distillation of the heavy product is shown schematically in Figure I. The distillation was done in two stages: 20-40mm Hg vacuum to remove a fraction distilling below 250°C, 0.2 to 1mm Hg vacuum to obtain all the other fractions: 250-350°C, 350-407°C, 407-450°C, 450-525°C, 525°C.

Procedure

Weigh 50-60g. of sample into the previously tared vacuum distillation flask. Seal the flask onto the apparatus using hi-temp-vac lubricant. Surround the traps with the proper coolant, start the vacuum and observe the flask content for signs of foaming. The pressure on the distillation assembly shall be lowered gradually to deaerate and minimize the foaming. When the equipment is air tight, apply a small flame into the flask. Adjust the burner so that the first drop of condensate will be collected after 10-15 minutes, at which point the temperature and pressure are noted and recorded. After the first fraction (250°C) is collected, the distillation is stopped, the U-tube manometer replaced by the McLeod gauge and the water condenser is replaced by a vacuum connecting tube to prevent plugging line by the higher boiling fractions which tend to solidify at room temperature.

As soon as vapor or refluxing liquid appears at the joint between the flask and the column, adjust the rate of heating so that the distillation is recovered at a constant rate (approx. 2ml/min.). Continue the distillation until either the maximum temperature (525°C equivalent) is reached, or the cracking occurs (sudden rise in pressure or excessive smoking) in which case note the temperature at which cracking occurs.

After a distillation is completed, the apparatus should be allowed to cool for about 10 minutes before admitting air into it in order to prevent the possibility of vapor self-ignition. Use liquid nitrogen in the vacuum flask surrounding the glass trap.

FIGURE 1.